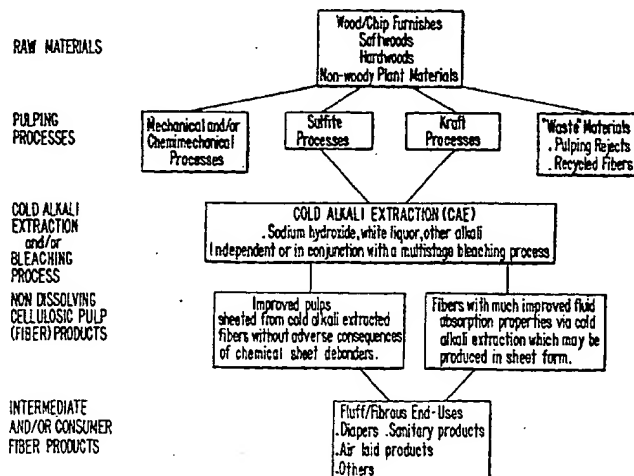




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(54) Title: COLD CAUSTIC EXTRACTION OF PULPS FOR ABSORBENT PRODUCTS



## (57) Abstract

An improved absorbency material for absorbency applications comprised of a cellulosic fibrous material wherein said cellulosic fibrous material such as pulp is a cold alkali solution treated material at a treatment temperature of less than about 50 °C; a process for improving absorbency and other characteristics of said pulp.

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## 1 COLD CAUSTIC EXTRACTION OF PULPS FOR ABSORBENT PRODUCTS

2

3 SPECIFICATION

4 This invention relates to pulps for absorbent products, more  
5 particularly this invention relates to novel use of modified pulps for absorbent  
6 products of household and hygienic uses such as diapers, incontinence and  
7 catamenial devices and the like and a method for preparing pulps of outstanding  
8 absorbency characteristics.

9 More particularly, this invention relates to the novel use of known  
10 technology - cold alkali extraction - to produce cellulosic pulps having altered and  
11 novel fiber properties desirable for end-use applications for absorbent and fluff pulp  
12 products.

13 Further, this invention relates to the production of the altered and  
14 novel pulps without addition of chemical additives such as sheet debonders. Still  
15 further this invention relates to the production of pulps having novel, desirable  
16 properties achieved without chemical modification steps such as cross-linking with  
17 chemical cross-linking agents.

18 Moreover, this invention relates to a novel use of a pulp product  
19 characterized and defined by its properties for suitable end uses of these pulps.  
20 Accordingly, novel pulp products can be obtained at reduced cost for the  
21 respective, economic effectiveness of these pulps when compared to pulps  
22 prepared by the prior art and suitable for the same purpose.

23 BACKGROUND FOR THE INVENTION

24 With the increasing prominence of disposable items, such as  
25 diapers, paper towels and the like, and in view of the widely prevalent use of  
26 absorbent tissues of various kinds, it has become important to obtain pulps of high  
27 absorbency, especially pulps that display high absorbency upon multiple re-  
28 wetting.

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1           Typically pulps that are used for hygienic absorption purposes such  
2 as baby diapers and the like are constructed with an outside "acquisition" layer,  
3 which is a layer of pulp of good bulking properties and good absorbency due to  
4 relative fiber stiffness. A bulky material will contain a high percentage of void  
5 spaces or pores. For an absorbent product, these pores are used to acquire,  
6 transport and store fluid. Longer, stiffer fibers make bulkier air laid webs with more  
7 pore volume. Fluids are more easily acquired and transported if pore volume or  
8 bulk is high. The "acquisition" layer is positioned between the baby's skin and the  
9 absorbent core of the diaper. An "acquisition" layer of proper characteristics and  
10 properties allows the liquid to pass quickly into the absorbent core upon repeated  
11 wettings and at the same time this layer transmits the liquid into the principal  
12 absorbent core that holds the liquid. In a similar manner, an incontinent or  
13 catamenial device may be constructed. Further, wound dressing material may be  
14 construed in a like manner. These devices are absorbency products which require  
15 pulps having intensive absorbency properties.

16           Still further, absorbent multi-ply papers such as household towels  
17 may be constructed of multiple layers or plies including a core layer and thus  
18 these plies may be tailored according to the use to which these goods are being  
19 subjected or for the purpose these are employed.

20           Products such as diapers when used with an outside "acquisition"  
21 layer and an interior principal absorbent core, are presently desirably constructed  
22 with the "acquisition" layer made from cross-linked pulps such as are illustrated by  
23 the following European Patent applications 0 427,316 A2 and 0 427,317 A2 all by  
24 Herron et al. and U.S. Patent no. 5,137,537 by Herron et al. assigned to Proctor &  
25 Gamble Co. Further, Canadian Patent application 2,035,402, by Kokko based on

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1 U.S. priority application 07/473,404 and assigned to James River Corp. likewise  
2 discloses such pulps.

3 Cross-linked pulps are typically prepared using formaldehyde-based  
4 compounds. More recently, polycarboxylic acids, particularly citric acid, have been  
5 shown to be effective cross-linking agents. Cross-linked fibers display excellent  
6 wet stiffness. The cross-links physically restrict the uptake of water into the fiber  
7 wall. By doing so, the fiber retains, better than conventional fiber, the  
8 characteristic stiffness of dry fibers. A web of cross-linked fibers, therefore, retains  
9 its bulk and pore volume when wet, which enhances fluid acquisition, especially  
10 with repeated wettings or insults. However, chemically cross-linked fibers are  
11 considerably more expensive than fibers which may be employed without any  
12 cross-linking. Moreover, pulps employed in prior art processes for cross-linking  
13 purposes are generally not available in sheeted form (rolls or bales of sheets).

14 Although pulps have been bleached under various alkaline  
15 conditions, bleaching schedules and bleaching treatment are by now those  
16 typically employed by prior art. Accordingly, a wide variety of such schedules are  
17 practiced -- for the most part employing at least one or more alkaline steps at fairly  
18 high temperatures. In such sequences it has also been known to employ caustic  
19 solutions at lower temperature and then the same solution is used to bring up the  
20 temperature to or greater than a boiling point of the solution as shown in Canadian  
21 Patent 578,573 entitled "Purification of Wood Pulp" granted June 30, 1959. In this  
22 patent the pulps so produced are used for dissolving pulps, i.e., making cellulose  
23 acetate and other chemical derivatives of cellulose. No description has been  
24 found concerning the improvements in absorbency, rewetting properties, stiffness  
25 of fibers, etc. as described herein for the pulps as used for the devices or products

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1 as illustrated herein. Moreover, the distinction between dissolving pulps and fluff  
2 pulp should also be noted.

3 **BRIEF DESCRIPTION OF THE DRAWINGS**

4 With reference to the Drawing herein:

5 Figure 1 is a plan view of a typical baby diaper;

6 Figure 2 is a cross-sectional view of the diaper shown in Figure 1,  
7 along lines 2-2 thereof; and

8 Figure 3 is a schematic self-explanatory presentation of the overall  
9 process/product improvements.

10 **BRIEF DESCRIPTION OF THE INVENTION**

11 It has now been found that cold alkali extraction (CAE) of pulps  
12 such as preferably obtained from coniferous and deciduous trees results in fibers  
13 that have advantageously and unexpectedly improved absorption properties.  
14 Pulps from other source materials may also be suitable (e.g., bagasse, straw, etc.).  
15 By the term "cold" it is meant a caustic treatment not to exceed 60°C but desirably  
16 at a temperature less than 50°C but preferably at a temperature between 15°C to  
17 40°C. Cold alkali extraction is synonymous with cold caustic extraction CCE when  
18 caustic is used as the alkali source. By the term "caustic" it is meant sodium  
19 hydroxide solutions newly made up or as a solution by-product in a pulp or paper  
20 mill operation e.g., hemi caustic white liquor, oxidized white liquor and the like.  
21 Further, ammonium hydroxide, and potassium hydroxide and the like may be  
22 employed. However, from a cost standpoint, the preferable caustic material is  
23 sodium hydroxide. Cold alkali extraction may be performed with additional  
24 chemicals added such as hydrogen peroxide, sodium hypochlorite, sodium  
25 borohydride, various surfactants, etc.

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1                   The cold caustic extraction is typically at a caustic strength in a  
2 range from about 3% to 25%, preferably from about 6% to 18%, at a pulp  
3 consistency from about 2% to 25% but desirably from 2% to 10% but preferably  
4 from 3% to 8%. Pulps for high rate, fast absorbing applications are preferably  
5 treated with cold caustic concentrations from 13% to 18%. A wide variety of pulps  
6 are suitable such as obtained by mechanical or chemi-mechanical, sulfite, kraft,  
7 pulping reject materials, organic solvent pulps, etc. Both softwood and hardwood  
8 species are useful. Softwood pulps are preferred. Among pulps those that have  
9 not been severely bleached are useful, for example pulps with high K Numbers  
10 (i.e., "potassium permanganate" number; a high K or Kappa Number signifies a  
11 relatively high residual lignin content for the pulp). The more heavily bleached pulp  
12 will be improved less and also requires a weaker alkali treatment. If the pulps are  
13 treated in the manner as it will be further disclosed in the specification herein, then  
14 the resulting fibers are such that these have good bulking, (i.e., "stiffness"  
15 properties) and thus have much improved absorption and rewetting properties  
16 making these pulps attractive for a number of uses. These pulps are not only  
17 characterized for their improved properties, such as by their ability to absorb and  
18 reabsorb water more quickly (than the standard untreated pulps) when subjected  
19 to multiple rewet tests, but also these pulps are useful for absorbent devices in the  
20 principal core for such device. In fact, the resulting improvements in the  
21 absorption properties are so significant that the products on an economical basis  
22 may readily compete with the more expensive prior art cross-linked products  
23 described in the above-identified patents.

24                   As mentioned above, the newly discovered pulp preparation has  
25 wide applicability to all types of pulp/fiber source materials and displays improved  
26 properties for each of the pulps/fibers (Figure 3). Upon a cold caustic treatment of

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1 the pulp/fibers, these show improved properties. For pulps prepared under  
2 different pulping conditions or processes such as sulfite, pre-hydrolyzed kraft  
3 process, conventional kraft process, organic solvent processes, or BCTMP  
4 (bleached chemi-thermal mechanical pulp), etc., the properties are invariably  
5 improved. The improved properties have been observed for all pulp and fiber  
6 types investigated. Differences, however, exist between pulps obtained from  
7 various wood species starting materials. Surprisingly, the improved properties are  
8 obtained regardless of the wood species which have been employed, for example,  
9 western hemlock, Douglas fir, Sitka spruce, Southern pine, Caribbean pine and the  
10 like. Other commercial softwood species (e.g., firs and spruces) and hardwood  
11 species (e.g., gums, oaks, eucalyptus, poplar, beech, aspen, etc.) yield  
12 advantageous properties as well.

13 In an advantageous embodiment, it seems that the best  
14 characteristics for the obtained pulp have been observed for pulps that are  
15 unbleached or only slightly bleached. Nevertheless, good results have also been  
16 observed with bleached or more highly bleached pulps. As a corollary, the more  
17 highly bleached is the pulp, the lower is the caustic strength that is required to  
18 obtain the desirable effects. However, the desirable absorbency effects are  
19 somewhat less when compared on a direct basis with cold caustic extracted pulps  
20 derived from high K Number unbleached pulps (i.e. the products derived from high  
21 K Number pulps are noticeably better).

22 With reference to an embodiment, and the drawing herein, a typical  
23 construction of a diaper is shown in Figures 1 and 2 therein. In Figure 1 the plan  
24 view of the diaper 3 in its open position shows the tabs 4 which are a part of a  
25 hook or loop component shown as 5 as its complementary element. Other diaper  
26 designs include tape-fasteners.

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1 In Figure 2 which shows in cross section along lines 2-2 of Figure 1  
2 the construction features of diaper 3 and with reference thereto from top to bottom  
3 each element in the cross section 2-2 is described as follows:

4 Item 11 is a thermally-bonded polypropylene coversheet. It is  
5 typically carded or spun. Item 12 is an airlaid cellulose acquisition layer. Elements  
6 13 are tissue webs of a typical basis weight of about 16 g/m<sup>2</sup>; the absorbent core  
7 is identified as 14 and is of a fluff and SAP (super absorbent polymer and pulp  
8 mixture of a basis weight of, 500 - 700 g/m<sup>2</sup>). The water barrier, which is a  
9 polyethylene sheet has been shown as 16.

While the above illustration has been for a diaper, other devices have been constructed in a similar manner. Further, for similar absorbent paper products, the pulps as modified herein show substantial improvement in product performance on an economical basis. Thus, products such as catamenial and incontinence devices are improved. Other candidate applications for which the presently disclosed pulps are suitable are paper towels, sanitary tissue papers, industrial wipes, etc. For the above applications, the modified pulps may be 100% of the improved pulps as constituent pulps in the product or may be used in the product in lesser quantities, i.e., used in various admixtures with other pulp, from about 100% to about 25%.

20 Test Procedures

21 Whenever these tests have been described, the industry employed  
22 standard test procedure for the test has been used. If any changes in the  
23 procedure have been made, the changes have been described specifically.

24 For purposes of evaluating the pulps obtained and described by the  
25 present disclosure as well as the invention herein, several tests were used to  
26 characterize the desirable fibrous end-use performance improvements resulting

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1 from the use of cold alkali extraction and to describe some of the analytical  
2 properties of the pulp products. Also, some of the terminology used in discussing  
3 the products in the examples has been defined.

4 A summary of these tests and definitions follows.

5 Pulp Analytical Properties

6 The K Number or Kappa test is carried out according to TAPPI  
7 Standard Method No. T-214-SU71. This test is a measure of residual lignin content  
8 in the pulp. The test indicates the relative degree of residual lignin content in a  
9 pulp as a consequence of pulping and the extent or severity of pulping.

10 Pulp brightness is a measure of pulp whiteness with 100% being the  
11 maximum. Pulp brightness data here are given as ISO brightness values in %.

12 The ISO brightness test is described in Tappi Method Number T272 (Handsheets)  
13 and T525 (Instrumentation) and uses as a measuring device a Datacolor 2000  
14 brightness meter.

15 Pulp Sheet Properties

16 Debonded pulps are fibrous end-use pulps (for example, fluff pulps)  
17 that have some chemical agent (debonder) added to inhibit interfiber bonding  
18 (addition of debonder results in a soft pulp sheet). The chemical agents,  
19 debonders, are commercial products added to fluff pulps during sheet forming  
20 which make the pulp sheet softer and easier to fluff. Debonders are closely related  
21 to fabric softeners chemically, and act in the same fashion. The force with which  
22 pulp fibers bond is measured indirectly by measuring the force (or energy)  
23 expended to debond or fluff a given pulp sheet.

24 The basis weight of a pulp sheet as described herein was  
25 determined on some of the products presented in the examples using a method  
26 based on TAPPI T220. A sheet of pulp, commonly 30 cm x 30 cm or of another

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1 convenient dimension, was weighed and then dried to determine the solids content  
2 (%) O.D.). The area of the sheet was then determined and the ratio of O.D. (oven  
3 dried) weight to a defined area was reported as the basis weight.

4           The caliper and sheet density were determined on some of the  
5 products presented in the examples using a method based on TAPPI T220. Sheet  
6 calliper was determined on test specimens from the basis weight test using a  
7 motor driven micrometer that met TAPPI T411 conditions. Sheet density was  
8 calculated as the ratio of basis weight to caliper.

9           Mullen strength and burst indexes were determined on some of the  
10 products presented in the examples using a method based on TAPPI T807. A TMI  
11 Monitor Burst 1000 was used to measure the hydrostatic pressure required to  
12 rupture (bursting strength) the pulp sheet when the pressure was increased at a  
13 controlled constant rate through a rubber diaphragm to a circular area 30.5 mm  
14 diameter. Mullen strength is recorded as kPa (kilo Pascals) at rupture, while burst  
15 index is the ratio of bursting strength to basis weight.

16           A Kamas Lab hammermill Model H-01-C was used to defiberize  
17 some of the products presented in the examples. Strips of pulp sheets 5 cm wide  
18 were fed into the hammermill, using 900 rpm motor speed, 50% feeder speed, and  
19 an 8 mm screen. In some cases, the energy required to defiberize the pulp sheet  
20 was recorded, and reported as W hr/kg of fluff, the energy of defiberization. Fluff  
21 was collected in a collection vacuum bag for further testing.

22           An M/K Formation Tester was used to measure the formation of  
23 pulp sheets for some of the samples presented in the examples. The formation is  
24 an expression of sheet uniformity. The M/K Formation Tester consists of a  
25 rotating glass drum containing a traveling light source. A pulp sheet is wrapped  
26 around the outer surface of the drum. The light from inside the drum shines

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1 through the sheet and strikes a detector outside the drum. During the test, the  
2 drum rotates while the internal light source and the external detector move  
3 together down the axial length of the drum. In this way, the amount of light which  
4 passes through the sheet is measured at several different locations. The variation  
5 in the amount of light which passes through the sheet from point to point on the  
6 sheet is used as a measure of the formation (uniformity of formation) of the sheet.

7 Weighted average fiber length (WAFL) and fiber coarseness were  
8 also measured for some of the products presented in the examples using a  
9 Kajaani FS-200 Fiber Analyzer.

#### 10 Fiber Property Performance Tests

11 SCAN testing of fluff pulp properties was carried out on some of the products  
12 presented in the examples. This test uses SCAN/PFI methodology (SCAN-C  
13 33:80) and test equipment to form a uniform fluff sample, and to measure its  
14 resiliency, fluid retention and rate of absorption. The fluff samples are conditioned  
15 for at least 2 hours under standard conditions ( $23 \pm 1^\circ\text{C}$  and  $50\% \pm 2\%$  relative  
16 humidity) prior to testing and are kept in the conditioning atmosphere throughout  
17 the test.

18 A cylindrical fluff sample ( $3.00 \pm 0.05$  g and 5 cm diameter) is  
19 prepared using special equipment. The height of the cylinder under a 260 g/1.3  
20 kPa load is measured and reported as resiliency. The sample is placed in contact  
21 with a water bath. The time required for the water to migrate vertically up the  
22 cylinder to the top is reported as absorption time. The fluid retention or absorption  
23 capacity per gram of sample is calculated by weighing the saturated fluff sample.

24 A fluff sample can also be subjected to simulated heat-aging  
25 artificially ( $105^\circ\text{C}$  for two hours) and tested by this method to determine effects of  
26 aging on fluff absorbent properties.

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1                   Dry classification of fluff pulp was carried out on some of the  
2 products presented in the examples. This test is a measure of fluff quality and the  
3 defiberization process. A Johnson Manufacturing Fluff Fiberization Measuring  
4 Instrument, Model 9010, was used to separate the fluff into three fractions based  
5 on particle size. During the test, fluff is pneumatically agitated to separate the  
6 fibers from each other and from the undefibered pulpsheet. A vacuum draws the  
7 initial fines and then the long fibers through a rotating sieve screen (16 mesh, 1.18  
8 mm opening, U.S.A. std. series). The initial fines also pass through a second  
9 screen, and accumulate in a dust bag. The long fibers (accepts) accumulate on a  
10 second screen (45 mesh, 0.36 mm opening, U.S.A. std. series).

11                   Pad integrity testing was carried out on some of the products  
12 presented in the examples. Pad integrity is a measure of the strength of the fiber  
13 network in fluffed pulps, and indicates how well the fluff will maintain pad integrity  
14 in a dry formed absorbent product. The method is based on PFI method of 1981,  
15 "Measurement of Network Strength in Dry, Fluffed Pulps". During the test, a  
16 cylindrical test pad of  $1.0 \pm 0.05$  gram and 50 mm diameter is prepared in a pad  
17 former. The test pad is placed in a burst chamber, which is then installed in a  
18 stress-strain apparatus. A burst-body is vertically forced through the test pad. The  
19 force required to rupture the fiber network in the test pad is reported as pad  
20 integrity.

21                   The potential of a fibrous pulp for use as an acquisition layer can be  
22 described, among other tests, by a multiple "insult" or rewetting test. The Multiple  
23 Insult - Absorption Testing procedure was carried out as follows. Pulp for  
24 comparison purposes are fiberized, then airfiltered into pads with a basis weight of  
25 about 200 g/m<sup>2</sup>. The pads are pressed at 200 psig for a period of two minutes  
26 then trimmed about 7 cm x 16 cm. The trimmed and densified pads are placed on

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1 top of a standard absorbent core, such as a disposable diaper, and covered by a  
2 single layer of conventional polypropylene coverstock. Fluid is introduced to the  
3 absorbent product through a cylinder permanently mounted to a weighted plate  
4 which applies a force of 0.1 psig to the absorbent product. A dam is used to  
5 control fluid flow to the absorbent product. A timer is started when the dam is  
6 removed and fluid begins to move into the absorbent product. The timer stops  
7 when all the fluid has been absorbed and the elapsed time recorded. Ninety  
8 seconds after the fluid is completely absorbed a stack of five preweighed blotter  
9 papers is placed on top and then a 1.0 psig load is applied to the absorbent  
10 product for a period of two minutes. The amount of fluid wetted back into the  
11 blotter paper is recorded. The procedure is repeated two times for a total of three  
12 wettings or "insults". The multiple "insult" test characterizes the readiness with  
13 which fibers absorb as well as reabsorbs a fluid.

14 DETAILED DESCRIPTION OF THE INVENTION  
15 AND EMBODIMENTS THEREOF  
16

17 As mentioned above, it has now been found that various pulps of  
18 diverse wood species prepared by diverse pulping and bleaching processes  
19 provide improvements in these pulps by displaying improved fiber and pulp sheet  
20 properties, e.g. absorbency results such as for an acquisition layer in baby  
21 diapers, etc. upon cold alkali extraction (CAE) or cold caustic extraction (CCE) of  
22 these pulps in the proper manner in the proper sequence when preparing these  
23 pulps, i.e., when treating the pulps. Relatively high strengths of sodium hydroxide  
24 solution are used ideally, 13% - 18% NaOH by weight for high absorbency, fast  
25 intensive absorbency applications and 5% to 15% for general absorbency  
26 application, preferably 6% to 10% for that purpose. By "cold caustic extraction"  
27 (CCE) is meant the treatment of pulp at a temperature less than 60°C, preferably  
28 less than about 40°C, with the above sodium hydroxide solutions. The process

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1 coextensive with the preparation of the novel pulps is being claimed as an  
2 improvement for the regime of the novel properties heretofore unrecognized in the  
3 art. The improvement thus also resides in a method for improving, e.g., the  
4 absorbency of the pulp, increasing the stiffness of fibers and other properties  
5 further described herein not heretofore known or recognized.

6               Moreover, it has been found as an embodiment that the appropriate  
7 "acquisition layer" absorbency performance can be established after adequate  
8 bleaching of high K number unbleached pulp has been carried out to obtain  
9 aesthetically acceptable brightness values for the pulps with slightly lower cold  
10 caustic treatment (e.g. 15% NaOH versus 18% NaOH for unbleached pulps). At  
11 the lower concentration of cold caustic solution bleached pulps are obtained which  
12 are nearly as good as pulps obtained from CCE of the high K number unbleached  
13 pulps themselves.

14               Accordingly, it has been found that a specific desirable pulp product  
15 regime exists based on the process employed and the selection of various product  
16 or fiber criteria as will be further described herein. For example, the absorbency  
17 relationships make the pulps in the characterized pulp product regime especially  
18 useful because the pulps and their use can now be readily delineated from the  
19 regime of unattractive uses and pulps not possessing the attractive characteristics.  
20 Moreover, the relationships within this novel regime of other desirable properties  
21 has been established so as to delineate with great precision the claimed regime of  
22 the novel properties and the technique and process coextensive therewith.

23               Still further, while cold caustic treatment has been known for high  
24 quality dissolving pulps as discussed above, e.g. to make alpha cellulose and  
25 some industrial product pulps, such treatment as correlated to the fiber and pulp  
26 sheet property variables listed above is novel with respect to pulps useful such as

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1 for absorbent pulps e.g. for an acquisition layer for products *inter alia* diapers,  
2 incontinent and catamenial devices, etc. including absorbent core materials for  
3 these.

4 Added and further benefits will appear from the following examples  
5 and the illustrative embodiments. The examples are merely for the purpose of  
6 illustration and are not intended to limit the scope of the invention.

7 Example 1. Improved Pulp Sheet Defiberization:  
8 Debonders vs. Cold Alkali Extraction, Kraft  
9 Southern Pine Pulp

10  
11 Cellulosic pulp is commonly manufactured for fluff and other fibrous  
12 end-use in dried, sheeted form. The pulp manufacturer operates the pulp machine  
13 to form the sheet from an aqueous suspension of fibers; the sheet once formed is  
14 dried to remove about 90% of the moisture. Large rolls of dried, sheeted pulp are  
15 produced off the dry end of the pulp machine. These are typically cut into smaller  
16 size rolls and/or bales of sheets for distribution to end-use customers.

17 It is an advantage that the dried, sheeted pulp defibers easily and  
18 uniformly without damage to the individual fibers for those pulp grades being used  
19 in various fibrous end-use applications. For example, a fluff pulp will be converted  
20 by the end-user from the dried, sheeted pulp to a pad of "fluffed" fibers by  
21 mechanical action such as is supplied by a hammermill or other attrition mill.  
22 Chemical agents, debonders, are sometimes added to the pulp during sheet  
23 formation to inhibit interfiber bonding, which results in softer, more easily defibered  
24 sheets.

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## EXAMPLE 1, TABLE I-1

1			
2			
3	SAMPLE DESIGNATION	A-1i	B-1i
4	SAMPLE DESCRIPTION		
5	Processing	Non-debonded Standard Process	Debonded Standard Process
6	Wood Species	Southern pine blend . . . . . >	
7			
8	Pulping Process	Kraft . . . . . >	
9			
10	Sheet Debonder Used (?)	No	Yes
11			
12	COLD ALKALI EXTRACTION	Not Used	Not Used
13			
14	PULP ANALYTICAL PROPERTIES		
15	ISO Brightness, %	88.5	89.4
16			
17	SHEET PHYSICS		
18	Basis Wt. (g/m <sup>2</sup> )	640	634
19	Caliper (mm)	1.36	1.36
20	Density (g/cm <sup>3</sup> )	0.47	0.47
21	Mullen (kPa)	1113	417
22	Burst Index (kPa•m <sup>2</sup> /g)	1.74	0.66
23			
24	KAMAS FLUFF CHARACTERISTICS		
25	Resiliency (cm)	4.1	3.7
26	Fluid Retention (g/g)	13.4	11.4
27	Absorption Time (s)		
28	Control	3.5	7.9
29	Heat-Aged	4.4	8.9
30	Dry Classification (wt. %)		
31	Accepts	84.4	91.0
32	Knots	13.3	6.8
33	Fines	2.4	2.2
34	Pad Integrity (N)	7.2	7.0
35			

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## EXAMPLE 1, TABLE II-1

1			
2			
3	SAMPLE DESIGNATION	<u>A-1ii</u>	<u>B-1ii</u>
4	SAMPLE DESCRIPTION		
5	Processing	Standard Process (Non-debonded)	Cold Alkali Extraction Process
6			
7	Wood Species	Southern pine blend----->	
8			
9	Pulping Process	Kraft----->	
10			
11	Sheet Debonder Used (?)	No	No
12			
13	COLD ALKALI EXTRACTION	Not Used	Used
14	Alkali Used		NaOH
15	Solution Strength, %		7.5
16	Temperature, °C		35
17	Time, H:M		10
18	Consistency, %		3
19			
20	PULP ANALYTICAL PROPERTIES		
21	ISO Brightness, %	88.6	89.6
22			
23	SHEET PHYSICS		
24	Basis Wt. (g/m <sup>2</sup> )	644	666
25	Caliper (mm)	1.12	1.17
26	Density (g/cm <sup>3</sup> )	0.57	0.57
27	Mullen (kPa)	1494	829
28	Burst Index (kPa•m <sup>2</sup> /g)	2.32	1.25
29			
30	KAMAS FLUFF CHARACTERISTICS		
31	Resiliency (cm)	4.1	3.6
32	Fluid Retention (g/g)	12.9	13.2
33	Absorption Time (s)		
34	Control	3.3	2.9
35	Heat-Aged	4.9	4.6
36	Dry Classification (wt. %)		
37	Accepts	81.2	91.4
38	Knots	15.9	6.0
39	Fines	2.9	2.7
40	Pad Integrity (N)	7.2	7.4
41			

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EXAMPLE 1, TABLE III-1

1				
2				
3	SAMPLE DESIGNATION	<u>A-1iii</u>	<u>B-1iii</u>	<u>C-1iii</u>
4	SAMPLE DESCRIPTION			
5	Processing	Non-debonded Standard Process	Debonded Standard Process	Cold Alkali Extraction Process
6	Wood Species	Southern pine blend----->		
7				
8	Pulping Process	Kraft----->		
9				
10	Sheet Debonder Used (?)	No	Yes	No
11				
12	COLD ALKALI EXTRACTION	Not Used	Not Used	Used
13	Alkali Used			NaOH
14	Solution Strength, %			8.5
15	Temperature, °C			35
16	Time, H:M			0:10
17	Consistency, %			3
18				
19	PULP ANALYTICAL PROPERTIES			
20	ISO Brightness, %	86.6	88.4	88.9
21				
22	SHEET PHYSICS			
23	Basis Wt. (g/m <sup>2</sup> )	642	639	652
24	Caliper (mm)	1.36	1.30	1.33
25	Density (g/cm <sup>3</sup> )	0.48	0.49	0.49
26	Mullen (kPa)	1126	716	770
27	Burst Index (kPa•m <sup>2</sup> /g)	1.75	1.12	1.18
28				
29	KAMAS FLUFF CHARACTERISTICS			
30	Resiliency (cm)	4.1	3.9	3.6
31	Fluid Retention (g/g)	13.5	12.4	12.5
32	Absorption Time (s)			
33	Control	3.3	7.1	2.5
34	Heat-Aged	4.3	7.7	3.0
35	Dry Classification (wt. %)			
36	Accepts	84.2	87.4	94.6
37	Knots	13.1	10.3	3.6
38	Fines	2.7	2.3	1.8
39				

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1                   Table I-1 of Example 1 compares some of the conventionally  
2 prepared sheet property and fluff characteristics of "non-debonded" bleached  
3 kraft Southern pine pulp (Sample A-1i) to debonded (with chemical debonder  
4 added) bleached kraft Southern pine pulp (Sample B-1i). The pulp sheet products  
5 were produced on a commercial pulp machine. The sheet properties (or sheet  
6 physics) as well as characteristics of the fluffed fiber were tested as produced by a  
7 small scale hammermill. The data given are averages of several tests on pulp from  
8 several production runs. A description of the terms and tests has been given  
9 above.

10                   In comparison to the standard process non-debonded, sheeted  
11 pulp, the standard process debonded sheeted pulp is softer (weaker) indicated by  
12 the substantially lower pulp sheet Mullen strength as well as by the lower burst  
13 index. Note that the characteristics of the fluff from the debonded pulp are poorer  
14 as indicated by the lower resiliency, lower fluid retention, and increased (slower)  
15 absorption times compared to those of the standard pulp fluff. The dry  
16 classification data of the fluffed fibers from the debonded pulp do indicate,  
17 however, that better or more uniform defiberization was achieved (higher accepts,  
18 lower knots). The fluff pad integrity was equivalent for both types of pulp.

19                   In Table II-1 of Example 1, pulp produced by the process of this  
20 invention, cold alkali extraction (Sample B-1ii) is compared to standard process  
21 pulp (Sample A-1ii). Both types of pulp were pulped by the kraft process from a  
22 Southern pine chip furnish and were bleached to similar brightness using standard  
23 chemicals/conditions of chlorine, chlorine dioxide, sodium hydroxide and sodium  
24 hypochlorite. The data given are mean data for several samples tested during  
25 standard production and trial production periods. The conditions used during the

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1 cold alkali extraction averaged about 7.5% NaOH solution strength, at about 35°C  
2 for about 10 minutes at a pulp consistency of about 3%.

3 Note that the cold alkali extraction processed pulp sheet was softer  
4 (about 45% lower in Mullen strength and in burst index) compared to the standard  
5 process pulp sheet. Also, the dry classification data of the fluff produced upon  
6 small scale fluffing showed improvements in the greater percent accepts and in the  
7 percent lower "knots" which indicates improved defiberability relative to the  
8 standard process pulp sheet. In these respects, the effect of the cold alkali  
9 extraction process on the pulp and fluff properties relative to the standard, non-  
10 debonded pulp were similar to the effects of the use of a chemical pulp sheet  
11 debonder relative to standard process pulp (Table I-1, Example 1) and, in fact, the  
12 novel pulps showed improved absorption properties. However, the cold alkali  
13 extraction process did not result in any negative consequences on fluff absorption  
14 times as does the use of a debonder. The fluid retention of the fluff from the cold  
15 alkali extraction processed pulp was equivalent to that of the standard pulp (Table  
16 II-1, Samples A-1ii and B-1ii).

17 Note that the percentage improvement was greater in the weight  
18 percent accepts (and in lower knot content) in the fluff from the cold alkali  
19 extraction process pulp compared to the fluff from standard pulp (Table II-1) than  
20 the comparable improvement associated with the use of a sheet debonder (Table  
21 I-1). The dry classification accepts were 12% greater and the knots 62% reduced  
22 for the cold alkali extraction process pulp relative to its control standard process  
23 pulp, whereas accepts for the debonded pulp were increased by only 8% with knot  
24 content reduced only 49% relative to its standard process control pulp. Also,  
25 these relative improvements were achieved by the cold alkali extraction processed  
26 pulp from a sheet that was actually somewhat harder than the debonded pulp

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1 sheet (829 Mullen strength/1.25 burst index vs. 417 Mullen strength/0.66 burst  
2 index).

3               The data presented in Table III-1, of Example 1 compare mill  
4 production of both debonded and non-debonded standard pulps with trial  
5 production of cold alkali extraction pulp. All pulp types (Samples A-1iii, B-1iii, and  
6 C-1iii) were produced from a Southern pine chip blend furnish by the kraft pulping  
7 process. All pulp types were bleached with chlorine dioxide, sodium hydroxide,  
8 oxygen and/or hydrogen peroxide to the brightness level indicated. The cold  
9 alkali extraction conditions achieved averaged about 8.5% NaOH solution strength,  
10 at about 35°C for about 10 minutes applied to a pulp slurry at 3% consistency.  
11 About 0.2% H<sub>2</sub>O<sub>2</sub> (O.D. --oven dried-- pulp basis) had been added during the cold  
12 alkali extraction.

13               Note that the debonded pulp and the cold alkali extracted pulp  
14 sheet were produced at approximately the same Mullen strength and burst index,  
15 with both of these indicators of sheet hardness being substantially reduced for  
16 either type of treated pulp, debonded or cold alkali extracted relative to the  
17 standard process control. Again, the fluff properties for the cold alkali extracted  
18 pulp showed some similarities to the debonded standard process pulp: resiliency  
19 and fluid retention were directionally lower for both relative to the standard pulp  
20 fluff. But the absorption times for fluff from the cold alkali extracted pulp were  
21 better (faster) than for fluff from the debonded pulp or the standard process pulp.  
22 Fluff dry classification weight percentage accepts and percentage knots were  
23 directionally better for fluff from the trial cold alkali extraction pulp.

24               Thus, the use of cold alkali extraction resulted in advantages not  
25 found with the standard process pulp and/or not expected from known technology

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1 of applying sheet debonders to standard process pulp as a means of "softening"  
2 the pulp sheet.

3                   Example 2. Cold Alkali Extraction for Fiber Property  
4                   Improvement, Prehydrolyzed Kraft Southern Pine Pulp

5  
6                   The data presented in Tables I-2 and II-2 of Example 2 illustrate the  
7 pulp sheet and fiber property improvements which occurred when cold alkali  
8 extraction was applied to pulps cooked from a Southern pine furnish by a  
9 prehydrolyzed kraft process. The prehydrolyzed kraft process is a two-stage  
10 pulping process, in which the raw material furnish is treated first under a mildly  
11 acidic condition (pH of about 3-4), followed by an alkaline stage which is basically  
12 the kraft cook illustrated in Example 1.

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EXAMPLE 2, TABLE I-2

1	EXAMPLE 2, TABLE I-2		
2	SAMPLE DESIGNATION	<u>A-2i</u>	<u>B-2i</u>
3	SAMPLE DESCRIPTION		
4			
5	Processing	Non-debonded Standard Process	Cold Alkali Extraction Process
6			
7	Wood Species	Southern pine blend—————>	
8			
9	Pulping Process	Prehydrolyzed kraft—————>	
10			
11	Sheet Debonded Used (?)	No	No
12			
13	COLD ALKALI EXTRACTION	Not Used	Used
14	Alkali Used		NaOH
15	Solution Strength, %		15
16	Temperature, °C		25
17	Time, H:M		0:10
18	Consistency, %		3
19	PULP ANALYTICAL PROPERTIES		
20	ISO Brightness, %	89.4	86.7
21	SHEET PHYSICS		
22	Basis Wt. (g/m <sup>2</sup> )	652	818
23	Caliper (mm)	1.32	1.28
24	Density (g/cm <sup>3</sup> )	0.50	0.64
25	Mullen (kPa)	1154	716
26	Burst Index (kPa•m <sup>2</sup> /g)	1.77	0.88
27	KAMAS FLUFF CHARACTERISTICS		
28	Resiliency (cm)	3.8	4.0
29	Fluid Retention (g/g)	13.2	13.1
30	Absorption Time (s)		
31	Control	3.4	3.4
32	Heat-Aged	4.2	6.1
33	Dry Classification (wt. %)		
34	Accepts	95.0	97.3
35	Knots	1.8	0.7
36	Fines	3.2	2.0
37	Pad Integrity (N)	6.8	7.3
38	MULTIPLE INSULT ABSORPTION TEST		
39	Absorption times, seconds		
40	1st insult	4.3	3.1
41	2nd insult	30.6	23.1
42	3rd insult	45.1	31.1
43			

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EXAMPLE 2, TABLE II-2			
		<u>A-2ii</u>	<u>B-2ii</u>
1			
2	SAMPLE DESIGNATION		
3	SAMPLE DESCRIPTION		
4			
5	Processing	Non-debonded Standard Process	Cold Alkali Extraction Process
6			
7	Wood Species	Southern pine blend—————>	
8			
9	Pulping Process	Prehydrolyzed kraft—————>	
10			
11	Sheet Debonded Used (?)	No	No
12			
13	COLD ALKALI EXTRACTION	Not Used	Used
14	Alkali Used		NaOH
15	Solution Strength, %		10
16	Temperature, °C		25
17	Time, H:M		0:10
18	Consistency, %		3
19			
20	PULP ANALYTICAL PROPERTIES		
21	ISO Brightness, %	90.5	87.2
22			
23	SHEET PHYSICS		
24	Basis Wt. (g/m <sup>2</sup> )	847	897
25	Caliper (mm)	1.05	1.16
26	Density (g/cm <sup>3</sup> )	0.81	0.77
27	Mullen (kPa)	1090	671
28	Burst Index (kPa•m <sup>2</sup> /g)	1.29	0.75
29	Kamas Energy (wh/kg)	116	83
30			
31	KAMAS FLUFF CHARACTERISTICS		
32	Resiliency (cm)	3.8	3.7
33	Fluid Retention (g/g)	14.1	13.9
34	Absorption Time (s)		
35	Control	4.2	3.8
36	Heat-Aged	5.6	5.1
37	Dry Classification (wt. %)		
38	Accepts	95.3	95.4
39	Knots	1.1	1.4
40	Fines	3.6	3.2
41	Pad Integrity (N)	8.1	6.1
42			

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1                   Sample A-2i in Table I-2 is a prehydrolyzed kraft Southern pine pulp  
2   bleached with the conventional bleaching agents of chlorine, chlorine dioxide,  
3   hypochlorite and/or hydrogen peroxide and/or oxygen and sodium hydroxide to  
4   the ISO Brightness level indicated. Sample B-2i in Table I-2 is a similarly  
5   prehydrolyzed kraft pulp, similarly bleached to the brightness indicated prior to  
6   cold alkali extraction.

7                   The B-2i sample's processing included the cold alkali extraction  
8   process under the conditions listed (average conditions used). Both the A-2i and  
9   B-2i samples were produced in a mill scale facility during production and trial runs,  
10   respectively.

11                  As for the bleached Southern pine kraft pulp discussed in Example  
12   1, the use of cold alkali extraction resulted in a softer pulp sheet (*i.e.*, of lower  
13   Mullen strength and lower burst index). Note that the higher basis weight and  
14   density at which the B-2i sample pulp was produced should have had a negative  
15   impact on these sheet properties. In addition, the resiliency and fluid retention of  
16   the cold alkali extracted Sample B-2i were equivalent to those of Sample A-2i and  
17   the dry fluff classification results showed some improvement for Sample B-2i.  
18   Kamas fluff absorption times were similar (slightly longer for Sample B-2i upon  
19   heat-aging), but the specialized multiple insult absorption tests (described above)  
20   showed that cold alkali extraction improved the absorption properties of the  
21   resulting fiber.

22                  The data presented in Table II-2 also compare bleached  
23   prehydrolyzed kraft Southern pine pulps (pulping conditions were more severe  
24   than those used for the samples in Table I-2 of this example).

25                  Sample A-2ii was produced without, Sample B-2ii with cold alkali  
26   extraction. Both types were also produced in a mill-scale facility and used

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1 common bleaching techniques to reach the brightness levels indicated. The  
2 sodium hydroxide solution strength used was lower than that used for the samples  
3 described in Table I-2 of this example, 10% vs. 15%.

4 Comparison of sheet property data again shows the sheet softening  
5 effects resulting from the use of cold alkali extraction: lower Mullen strength, lower  
6 burst index. Also, a Kamas energy parameter (see description above) was  
7 recorded to describe the relative ease of defibering the pulp sheet during the  
8 fluffing operation in the laboratory. The cold alkali extracted pulp was fluffed more  
9 easily (with less energy input). Fluff absorptions were slightly faster for the cold  
10 caustic extracted Sample B-2ii. Dry classification of the fluffed fibers were  
11 equivalent for both samples as were resiliency and fluid retention. However, fluff  
12 pad integrity (see description above) was poorer for the cold caustic extracted  
13 Sample B (this was not the case with Sample B-2ii compared to Sample A-2i in  
14 Table I-2 of this example).

15 Example 3. Cold Alkali Extraction Process:  
16 Variable Solution Strength

17  
18 From the data given in Tables I-3 through VI-3 of this example, it is  
19 apparent that no one set of cold alkali extraction process conditions will result in  
20 exactly the same consequences on every type of pulp. Raw material/furnish used,  
21 pulping process used, and the position of cold alkali extraction within a bleaching  
22 sequence have consequences on what may be the optimum conditions for each  
23 type of sample. Secondly, it appears that cold alkali extraction conditions can be  
24 selected to enhance some of the fiber properties of the resulting pulp but at the  
25 expense of others. Not all fibrous end-uses require improvements in the same  
26 properties, thus this apparent versatility of cold alkali extraction conditions might  
27 be used to tailor pulp fibers for various fibrous end-use products and/or  
28 customers. These points will be discussed in this and subsequent examples.

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## EXAMPLE 3, TABLE I-3

1				
2				
3	SAMPLE DESIGNATION	A-3i	B-3i	C-3i
4	SAMPLE DESCRIPTION			
5	Processing	Non-debonded Standard process	Cold Alkali Extraction Process	
6				
7	Wood Species	Southern pine blend		
8				
9	Pulping Process	Kraft		
10	K Number, mL	18		
11				
12	COLD ALKALI EXTRACTION	Not Used	Used	
13	Alkali Used	(Control)	NaOH	NaOH
14	Solution Strength, %		7	12
15	Temperature, °C		35	35
16	Time, H:M		0:15	0:15
17	Consistency, %		3	3
18				
19	PULP ANALYTICAL PROPERTIES			
20	ISO Brightness, %	83.3	92.2	91.4
21				
22	SHEET PHYSICS			
23	Basis Wt. (g/m <sup>2</sup> )	725	737	691
24	Caliper (mm)	1.75	2.14	2.41
25	Density (g/cm <sup>3</sup> )	0.41	0.35	0.29
26	Mullen (kPa)	1179	527	70
27	Burst Index (kPa•m <sup>2</sup> /g)	1.63	0.72	0.10
28				
29	KAMAS FLUFF CHARACTERISTICS			
30	Resiliency (cm)	3.8	3.9	3.7
31	Fluid Retention (g/g)	13.3	12.2	14.6
32	Absorption Time (s)			
33	Control	3.7	2.9	3.9
34	Heat-Aged	4.3	3.6	4.1
35	Dry Classification (wt. %)			
36	Accepts	97.5	98.5	89.0
37	Knots	1.2	0.1	8.6
38	Fines	1.3	1.4	2.4
39	Pad Integrity (N)	6.7	6.4	5.3
40				
41				

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1	EXAMPLE 3, TABLE II-3				
2	SAMPLE DESIGNATION	<u>A-3ii</u>	<u>B-3ii</u>	<u>C-3ii</u>	<u>D3ii</u>
3	SAMPLE DESCRIPTION				
4					
5	Processing	Non-debonded Standard Process	Cold Alkali Extraction Process—>		
6					
7	Wood Species	Southern pine blend—————>			
8					
9	Pulping Process	Kraft—————>			
10	K Number, mL	18—————>			
11					
12	COLD ALKALI EXTRACTION				
13	Alkali Used	None (water)	NaOH—————>		
14	Solution Strength, %	0	3	7	14
15	Temperature, °C	35—————>			
16	Time, H:M	0:10—————>			
17	Consistency, %	3—————>			
18					
19	PULP ANALYTICAL PROPERTIES				
20	ISO Brightness, %	87.9	90.3	92.1	92.0
21					
22	SHEET PHYSICS				
23	Basis Wt. (g/m <sup>2</sup> )	711	677	676	691
24	Caliper (mm)	1.87	2.03	2.06	2.79
25	Density (g/cm <sup>3</sup> )	0.38	0.34	0.33	0.26
26	Mullen (kPa)	1189	1005	501	83
27	Burst Index (kPa•m <sup>2</sup> /g)	1.67	1.48	0.75	0.12
28	Kamas Energy (wh/kg)	109.5	99	92.7	—
29					
30	KAMAS FLUFF CHARACTERISTICS				
31	Resiliency (cm)	3.8	3.5	3.3	3.3
32	Fluid Retention (g/g)	11.8	11.3	11.2	13.2
33	Absorption Time (s)				
34	Control	4.1	2.8	2.6	4.0
35	Dry Classification (wt. %)				
36	Accepts	94.6	94.3	94.0	75.9
37	Knots	2.0	2.7	3.2	21.1
38	Fines	3.4	3.0	2.8	3.1
39	Pad Integrity (N)	7.4	6.4	5.7	4.2
40					

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EXAMPLE 3, TABLE III-3

	A-3iii	B-3iii	C-3iii	D-3iii	E-3iii
1					
2					
3	SAMPLE DESIGNATION				
4	SAMPLE DESCRIPTION				
5	Processing				
6	Cold Alkali Extraction Process				
7	Wood Species				
8	Southern pine blend				
9	Pulping Process				
10	K Number, mL				
11	18				
12	COLD ALKALI EXTRACTION				
13	Alkali Used				
14	Solution Strength, %				
15	Temperature, °C				
16	Time, H:M				
17	Consistency, %				
18	Sodium hydroxide (NaOH)				
19	7.0 11.0 13.1 15.1 18.2				
20	35°C				
21	0:15				
22	3				
23	PULP ANALYTICAL PROPERTIES				
24	ISO Brightness, %				
25	88.0 88.3 85.3 85.3 85.7				
26	MULTIPLE INSULT ABSORPTION TESTS				
27	Absorption Times, seconds				
28	1st insult				
29	7.6 6.6 6.8 6.2 6.5				
	37.6 26.2 25.1 21.8 22.4				
	56.6 44.9 36.5 35.0 33.0				

		EXAMPLE 3, TABLE IV-3					
		A-3iv	B-3iv	C-3iv	D-3iv	E-3iv	F-3iv
SAMPLE DESIGNATION							
SAMPLE DESCRIPTION							
Processing	Unbleached Pulp Before Cold Alkali Extraction	Unbleached Pulp After Cold Alkali Extraction					
Wood Species	Southern pine blend						
Pulping Process	Kraft						
K Number, mL	8.2						
COLD ALKALI EXTRACTION	Not Used	Used					
Alkali Used	(Control)	NaOH					
Solution Strength, %		6	9	12	15	18	
Temperature, °C		30°C					
Time, H:M		0:15					
Consistency, %		3.0					
MULTIPLE INSULT ABSORPTION TEST							
Absorption times, seconds							
1st insult	8.9	8.2	6.7	7.5	7.4	8.3	
2nd insult	43.8	43.7	30.7	32.8	29.6	31.7	
3rd insult	64.5	68.5	51.2	48.3	50.0	46.3	

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EXAMPLE 3, TABLE V-3

	SAMPLE DESIGNATION	A-3v		B-3v	
		Standard Process (Non-debonded)		Cold Alkali Extraction	
1					
2	SAMPLE DESIGNATION				
3	SAMPLE DESCRIPTION				
4	Processing				
5	Wood Species	Southern pine blend			
6	K Number, mL	12.4			
7	COLD ALKALI EXTRACTION	Not Used		Used	
8	Alkali Used	(Control)		NaOH	
9	Solution Strength			6	
10	Temperature, °C			28	
11	Time, H:M			0:15	
12	Consistency, %			3	
13	PULP ANALYTICAL PROPERTIES				
14	ISO Brightness, %	84.8		84.8	
15	SHEET PHYSICS				
16	Basis Wt. (g/m <sup>2</sup> )	644		599	
17	Caliper (mm)	1.33		1.40	
18	Density (g/cm <sup>3</sup> )	0.53		0.44	
19	Mullen (kPa)	1656		834	
20	Burst Index (kP•m <sup>2</sup> /g)	2.63		1.44	
21	Kamas Energy (wh/kg)	69.5		47.9	
22	KAMAS FLUFF CHARACTERISTICS				
23	Resiliency (cm)	4.1		3.7	
24	Fluid Retention (g/g)	12.0		11.8	
25	Absorption Time (s)				
26	Control	3.2		2.9	
27	Heat-Aged	4.8		4.5	
28	Dry Classification (wt. %)				
29	Accepts	86.6		88.7	
30	Knots	11.8		9.3	
31	Fines	1.6		1.9	
32	Pad Integrity (N)	7.1		7.0	
33					

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1	EXAMPLE 3, TABLE VI-3					
2	SAMPLE DESIGNATION	A - 3vi	B - 3vi	C - 3vi	D - 3vi	E - 3vi
3	SAMPLE DESCRIPTION					
4	Processing	Standard	Cold Alkali Extraction Process			
5		Process				
		(non-				
		debonded)				
6	Wood Species	Southern hardwood blend				
7	Pulping Process	Kraft				
8	K Number, mL	11.0				
9	COLD ALKALI EXTRACTION					
10	Alkali Used	None	Sodium hydroxide			
		(control)				
11	Solution Strength, %	—	9	12	15	18
12	Temperature, °C	30°C				
13	Time, H:M	0:15				
14	Consistency, %	3				
15	PULP ANALYTICAL PROPERTIES					
16	ISO Brightness, %	31.5	43.2	38.6	37.6	37.8
17	KAMAS FLUFF CHARACTERISTICS					
18	Resiliency (cm)	3.4	2.9	2.9	3.0	2.7
19	Fluid Retention (g/g)	12.5	12.2	13.0	13.2	12.6
20	Adsorption Time (s)					
21	Control	6.2	4.5	4.0	4.3	3.8
22	Heat-Aged	12.9	8.1	9.3	7.1	6.5
23	Dry Classification (wt. %)					
24	Accepts	93.2	93.7	91.6	91.5	90.4
25	Knots	0.7	0.6	0.6	0.7	0.5
26	Fines	6.1	5.7	7.6	7.8	9.1
27	Pad Integrity (N)	3.6	4.4	4.1	4.3	4.1
28	MULTIPLE INSULT ABSORPTION TEST					
29	Absorption times, seconds					
30	1st Insult	13.3	11.9	11.4	11.7	10.7
31	2nd Insult	55.3	46.1	46.8	45.8	45.9
32	3rd Insult	98.5	64.2	67.3	73.6	74.3

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1                   The data given in Table I-3 of this example cover laboratory  
2 experiments on bleached Southern pine kraft pulp. All Samples A-3i through C-3i  
3 were bleached to the brightness indicated with the common bleaching chemicals  
4 of chlorine dioxide, hydrogen peroxide and sodium hydroxide. The cold alkali  
5 extracted Sample B-3i and C-3i were additionally bleached with chlorine dioxide  
6 subsequent to cold alkali extraction (this contributed to the higher brightness levels  
7 of Samples B-3i and C-3i). The NaOH solution strength used in the cold alkali  
8 extraction of Sample B-3i was relatively low (7% compared to that used for Sample  
9 C-3i, 12%). These samples were sheeted (non-directional sheet on a laboratory  
10 sheet mold) and dried under the same standard conditions in the laboratory (but  
11 dried without restraint unlike a commercial pulp machine) so that the changes in  
12 pulp sheet and fiber/fluff properties measured reflect the cold alkali extraction  
13 process alone. Thus for Sample B-3i, cold alkali extraction resulted in fibers which  
14 formed a sheet which was fluffed into fibers with the following properties relative to  
15 Sample A-3i (non-cold caustic extraction): similar basis weight, higher caliper,  
16 lower density, lower Mullen strength and lower burst index; equivalent fluff  
17 resiliency but reduced fluid retention, faster absorption times and somewhat  
18 improved dry fluff classification profiles. For Sample C-3i (cold caustic extracted at  
19 12% NaOH solution strength) relative to Sample A-3i (non-cold caustic extracted)  
20 the comparison/contrast was as follows: slightly reduced basis weight, higher  
21 caliper, lower density, much lower Mullen strength and burst index (very weak  
22 sheet); equivalent fluff resiliency, higher fluff retention, similar Kamas fluff  
23 absorption times, and poorer dry fluid classification profile. Thus, if an end-use  
24 required a fiber of highest fluid retention, a higher caustic solution strength could  
25 be selected; to maximize accept fiber upon fluffing, a lower caustic strength would  
26 be preferable.

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1                   In Table II-3 of this Example 3, data are presented as additional data  
2 on bleached Southern pine kraft pulp, produced in the laboratory over a wider  
3 range of cold alkali extraction solution strengths. All samples A-3ii through D-3ii  
4 were bleached with the common bleaching agents of chlorine, chlorine dioxide,  
5 hydrogen peroxide and oxygen (prior to cold alkali extraction) to the ISO  
6 brightness levels indicated. Even at 3% NaOH solution strength, the pulp sheet  
7 can be somewhat softened without any large negative consequences on fluff  
8 products. However, cold caustic extraction at 14% NaOH solution strength appear  
9 to result in a pulp sheet that was fluffed with apparent negative consequences on  
10 dry classification and resiliency with no advantage in absorption time but with a  
11 slightly higher fluid retention.

12                   Results from a similar series of bleached prehydrolyzed kraft  
13 Southern pine pulps cold caustic extracted over a range of from 7 to 18% NaOH  
14 solution strength are presented in Table III-3. All samples A-3iii through E-3iii were  
15 bleached with chlorine, chlorine dioxide, sodium hypochlorite and sodium  
16 hydroxide prior to cold caustic extraction. The multiple insult absorption test  
17 results indicate that little benefit would be gained by using NaOH solution strength  
18 in the cold caustic extraction (under the temperature, time and consistency shown)  
19 greater than about 13% for this type of pulp, and that absorption times improve  
20 (decrease) progressively when using a 7% to 13% caustic solution.

21                   Similarly, data are given in Table IV-3 of this example which illustrate  
22 a levelling off of improvement of absorption times with increasing cold alkali  
23 extraction solution strengths (6-18%). Samples B-3iv through F-3iv were all cold  
24 alkali extracted from the starting material Sample A-3iv. No bleaching chemicals  
25 were used on any of the Samples A-3iv through F-3iv. Sample A-3iv was a  
26 Southern pine pulp cooked to very, very low K Number via a single stage

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1. conventional kraft cook in the laboratory. Multiple insult absorption times of the  
2 fluff fibers were improved with the use of a 9% NaOH solution strength with no  
3 further benefit being seen above the 9% strength. Thus a similar trend toward a  
4 levelling off of absorption times with increasing solution strength seen from  
5 application of cold alkali extraction was observed for this low K Number  
6 unbleached kraft Southern pine pulp as had been observed for the bleached  
7 prehydrolyzed kraft pulp sample series given in Table III-3 of this example. But the  
8 level of the absorption times at which each type of pulp absorbed and the solution  
9 strength at which the absorption time improved occurred differently with the type of  
10 pulp extracted.

11               For a very low K Number bleached kraft pulp, cold alkali extraction  
12 at relatively low NaOH solution strength has many advantages other than faster  
13 absorption times. Data are given in Table V-3 of this example which compare two  
14 samples (A-3v without and B-3v with cold alkali extraction) of pulp pulped to 12.4 K  
15 Number and bleached to 84.8% ISO Brightness. The cold caustic extraction  
16 conditions used for Sample B-3v were a relatively low solution strength of 6%, at  
17 28°C for 15 minutes at a pulp consistency of 3%. This cold alkali extraction was  
18 used prior to bleaching to the indicated brightness. Both types of pulp samples  
19 (data averages given for A-3v and B-3v) were bleached using chlorine dioxide,  
20 hydrogen peroxide and sodium hydroxide.

21               One of the largest drawbacks to pulping to low K Number by the  
22 kraft process is the negative consequences on fiber properties that result from the  
23 extensive pulping. Please compare the higher sheet Mullen strength and burst  
24 index of Sample A-3v, Table V-3 to those of the samples in Tables I-3 and II-3 of  
25 this Example 3. When the processing used on the low K Number pulp included  
26 cold alkali extraction (Sample B-3v, Table V-3), these disadvantages were

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1 overcome. In addition, the fluff characteristics of fibers from the B-3v sample low K  
2 Number pulp were equivalent to or better than the A-3v low K Number pulp sample  
3 (Table V-3, similar fluid retention, somewhat faster Kamas fluff absorption times  
4 and somewhat improved dry fluff classification profile).

5 In Table VI-3 of this example, data are presented for a series of  
6 unbleached kraft pulp cooked from a Southeastern United States blend of  
7 hardwoods (gums, oaks, etc.) to a low (11.0) K Number. Cold caustic NaOH  
8 solution strength was varied from 9 to 18% for Samples B-3vi through E-3vi;  
9 Sample A-3vi was the starting pulp prior to cold alkali extraction. No bleaching  
10 chemicals were used on these samples. The 9% NaOH solution strength appears  
11 to be optimum of the conditions studied for this type of pulp in the unbleached  
12 state: Kamas fluff and multiple insult absorption times are basically constant at 9%  
13 and higher solution strengths. Dry classification profile for the fluff deteriorate at  
14 solution strengths at or above 12%. For this type of hardwood pulp, even lower  
15 NaOH strength might prove to be more desirable. Note that hardwood pulps do  
16 not match softwood pulps in these types of fibrous property performance tests.  
17 However, cold alkali extraction can be used to improve some of these fiber/fluff  
18 properties to some extent for both types of furnishes.

19 Example 4. Sulfite Pulps: Debonders; Cold Alkali Extraction

20 Debonders can be used on sulfite process pulps as well as on kraft  
21 pulps. Data are presented in Table I-4, Example 4 for commercially available  
22 sulfite pulps. The pulping process used is acid sulfite (also known as acid  
23 bisulfite); both pulps are bleached to the brightness indicated by common  
24 bleaching agents such as chlorine, chlorine dioxide, hydrogen peroxide and  
25 sodium hydroxide. Data given are averaged from several tests of each type of  
26 production, non-debonded (A-4i) and debonded (B-4i).

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## EXAMPLE 4, TABLE I-4

1			
2			
3	SAMPLE DESIGNATION	<u>A-4i</u>	<u>B-4i</u>
4	SAMPLE DESCRIPTION		
5	Processing	Standard process non-debonded	Standard process debonded
6	Wood Species	Northwest U.S. softwood blend, predominantly Douglas fir →	
7			
8	Pulping Process	Sulfite →	
9			
10	Sheet Debonder Used (?)	No	Yes
11			
12	COLD ALKALI EXTRACTION	Not Used	Not Used
13			
14	PULP ANALYTICAL PROPERTIES		
15	ISO Brightness, %	91.8	91.2
16			
17	SHEET PHYSICS		
18	Basis Wt. (g/m <sup>2</sup> )	698	684
19	Caliper (mm)	1.25	1.34
20	Density (g/cm <sup>3</sup> )	0.56	0.51
21	Mullen (kPa)	479	279
22	Burst Index (kPa•m <sup>2</sup> /g)	0.69	0.41
23	Kamas Energy (wh/kg)	41.0	28.7
24			
25	KAMAS FLUFF CHARACTERISTICS		
26	Resiliency (cm)	3.7	3.6
27	Fluid Retention (g/g)	13.2	12.1
28	Absorption Time (s)		
29	Control	11.8	6.3
30	Heat-Aged	43.6	6.0
31	Dry Classification (wt. %)		
32	Accepts	77.9	87.5
33	Knots	16.0	7.1
34	Fines	6.1	5.4
35	Pad Integrity (N)	6.3	6.4
36			

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EXAMPLE 4, TABLE II-4

1					
2	SAMPLE DESIGNATION	<u>A-4ii</u>	<u>B-4ii</u>	<u>C-4ii</u>	<u>D-4ii</u>
3	SAMPLE DESCRIPTION				
4	Processing	Standard Process (non-debonded) Unbleached	Standard Process (non-debonded) Bleached	Cold Alkali Extraction Process Unbleached	Cold Alkali Extraction Process Bleached
5					
6	Wood Species	Southern pine blend—————>			
7					
8	Pulping Process	Acid sulfite—————>			
9	K Number, mL	34	28	34	28
10					
11	COLD ALKALI EXTRACTION	Not Used	Not Used	Used	Used
12	Alkali Used			NaOH	NaOH
13	Solution Strength, %			15	15
14	Temperature, °C			30	30
15	Time, H:M			0:15	0:15
16	Consistency, %			3	3
17					
18	PULP ANALYTICAL PROPERTIES				
19	ISO Brightness, %	--	86	--	86
20					
21	MULTIPLE INSULT ABSORPTION TEST				
22	Absorption times, seconds				
23	1st insult	13.2	7.9	6.8	8.1
24	2nd insult	36.3	37.2	20.9	27.3
25	3rd insult	45.1	63.5	30.1	38.6
26					
27					
28					
29					

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EXAMPLE 4, TABLE III-4

1			
2			
3	SAMPLE DESIGNATION	<u>A-4iii</u>	<u>B-4iii</u>
4	SAMPLE DESCRIPTION		
5	Processing	Standard Processing (non-debonded)	Cold Alkali Extraction Process
6	Wood Species	Caribbean pine	
7			
8	Pulping Process	Sulfite	
9	K Number	24	
10			
11	COLD ALKALI EXTRACTION	Not Used	Used
12	Alkali Used	Not Used	NaOH
13	Solution Strength, %		15.4
14	Temperature, °C		30
15	Time, H:M		0:37
16	Consistency, %		8
17			
18	PULP ANALYTICAL PROPERTIES		
19	ISO Brightness, %	87.8	77.5
20			
21	KAMAS FLUFF CHARACTERISTICS		
22	Resiliency (cm)	3.4	2.9
23	Fluid Retention (g/g)	12.1	13.0
24	Absorption Times (s)		
25	Control	2.8	3.5
26	Heat-Aged	4.4	4.4
27	Dry Classification (wt. %)		
28	Accepts	92.4	80.4
29	Knots	1.6	11.3
30	Fines	6.0	8.3
31			
32	MULTIPLE INSULT ABSORPTION TESTS		
33	Absorption Times, seconds		
34	1st insult	38.1	15.9
35	2nd insult	88.2	45.0
36	3rd insult	135.8	69.6
37			
38			
39			

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## EXAMPLE 4, TABLE IV-4

1			
2			
3	SAMPLE DESIGNATION	<u>A-4iv</u>	<u>B-4iv</u>
4	SAMPLE DESCRIPTION		
5			
6	Processing	Standard Process (non-debonded)	Cold Alkali Extraction
7			
8	Wood Species	Douglas fir	>
9			
10	Pulping Process	Sulfite	>
11	K Number, mL	26.6	>
12			
13	COLD ALKALI EXTRACTION	Not Used	Used
14	Alkali Used		NaOH
15	Solution Strength, %		15.4
16	Temperature, °C		30
17	Time, H:M		1:00
18	Consistency, %		13
19			
20	PULP ANALYTICAL PROPERTIES		
21	ISO Brightness, %	90.4	83.4
22			
23	KAMAS FLUFF CHARACTERISTICS		
24	Resiliency (cm)	3.5	2.5
25	Fluid Retention (g/g)	14.1	13.0
26	Absorption Time (s)		
27	Control	4.6	3.8
28	Heat-Aged	6.2	4.5
29	Dry Classification (wt. %)		
30	Accepts	87.8	65.4
31	Knots	6.6	26.6
32	Fines	5.6	8.0
33			
34	MULTIPLE INSULT ABSORPTION TEST		
35	Absorption times, seconds		
36	1st insult	47.2	28.9
37	2nd insult	103.3	59.9
38	3rd insult	137.9	77.2
39			

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1                   As in the Example 1, Table I-1, which illustrated the effects of  
2   debonders on bleached kraft pulp sheet properties, debonders can act to "soften"  
3   the softwood sulfite sheet. Note the lower Mullen strength, burst index and Kamas  
4   energy of Sample B-4i compared to Sample A-4i. There is also a similar trend  
5   toward lower fluid retention for the debonded pulp as was seen with the kraft pulp.  
6   The dry fluff classification profile is improved toward greater accepts, lower knots  
7   as was the case with the kraft pulp. However, sulfite pulps differ from kraft pulps  
8   in that the use of debonders improve absorption times for sulfite pulps (related to  
9   the differences in wood derived extractives in acid sulfite pulps). Note the long fluff  
10   absorption times for the non-debonded sulfite pulp.

11                  However, for sulfite pulps the use of cold alkali extraction can have  
12   additional advantages and can improve performance in the multiple insult test.  
13   Data are given in Table II-4 of this example which compare multiple insult tests for  
14   unbleached vs. bleached sulfite Southern pine pulps, processed with and without  
15   cold alkali extraction. Samples B-4ii and D-4ii were bleached with common  
16   bleaching agents such as chlorine, chlorine dioxide, hydrogen peroxide and  
17   sodium hydroxide. Samples A-4ii and C-4ii represent two high K Number  
18   unbleached Southern pine sulfite pulps. Sample C-4ii was cold alkali extracted  
19   using the conditions indicated in Table II-4 from Sample A-4ii; Sample D-4ii was  
20   cold alkali extracted from Sample B-4ii.

21                  These data indicate that cold alkali extraction can minimize  
22   differences in multiple insult performance of these Southern pine sulfite pulps  
23   apparent for unbleached vs. bleached pulp. Please compare the delta ( $\Delta$ ) for first  
24   absorption times for (A-4ii to B-4ii) of 5.3 seconds, but for delta first absorption  
25   times for (D-4ii to C-4ii) of 1.3 seconds; delta for third absorption times for (B-4ii to  
26   A-4ii) of 18.4 seconds, but only a delta of 8.5 seconds for the third absorption

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1 times for (D-4ii to C-4ii). Thus cold alkali extraction reduced the magnitudes of the  
2 differences in absorption times between unbleached and bleached pulps as well as  
3 improving the actual level at which both cold caustic extracted samples C and D  
4 performed (reduced all absorption times, further discussion of these samples is  
5 given in Example 5).

6 Data are presented in Tables III-4 and IV-4 of Example 4 for sulfite  
7 pulps which demonstrate the effects of using relatively high sodium hydroxide  
8 solution strengths for cold alkali extraction over longer retention time and at higher  
9 consistency than were used in the previous examples and tables. All four pulp  
10 samples (A-4iii and B-4iii, Table III, A-4iv and B-4iv, Table IV) were bleached using  
11 the common pulp bleaching chemicals of chlorine dioxide, hydrogen peroxide and  
12 sodium hydroxide. Acid sulfite pulping was used to cook a Caribbean pine chip  
13 furnish to 24 K Number (Table III-4); acid sulfite pulping was used to cook Douglas  
14 fir to about 27 K Number (Table IV-4). When cold alkali extraction was used (for  
15 the B samples of both these tables, i.e., B-4iii and B-4iv), it followed an initial  
16 bleaching stage treatment of the unbleached pulp with chlorine dioxide. Bleaching  
17 was continued following the cold alkali extraction to the brightness level indicated  
18 using chlorine dioxide, hydrogen peroxide and sodium hydroxide.

19 For the sulfite Caribbean pine pulp, the use of cold alkali extraction  
20 at 15.4% NaOH under the conditions listed resulted in fibers showing significant  
21 improvement in the multiple insult absorption test times (all three insult times were  
22 reduced about 50%). Kamas fluff characteristics were similar for the B-4iii sample  
23 compared to the A-4iii sample with the exception of a poorer dry classification  
24 profile for the cold caustic extracted sample B-4iii). This may be due to in  
25 optimally high NaOH solution strength for this type of pulp as was observed for  
26 some types of kraft pulps discussed under Example 4, and/or the higher

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1 consistency and/or longer time at which the relatively high concentration was used  
2 could be non-optimum.

3                   For the sulfite Douglas fir pulp of Table IV-4, the use of cold alkali  
4 extraction also markedly improved the multiple insult absorption test times; Kamas  
5 fluff absorption times were also faster but fluff resiliency and fluid retention  
6 appeared to be more greatly affected for Caribbean pine of Table III-4. The fluff  
7 dry classification profile was also poorer with cold alkali extraction of this Douglas  
8 fir sulfite pulp. Again, as with the sulfite Caribbean pine sample and with the kraft  
9 pulp examples discussed in Example 4, these cold alkali extraction conditions may  
10 be non-optimum for fiber properties other than absorption time improvement for  
11 this type of bleached sulfite softwood fiber.

12                   Example 5.   Kraft and Sulfite Southern Pine Pulps

13                   The following unbleached kraft pulp (Sample A-5i - Table I-5,  
14 Example 5) with a K Number of 30 was obtained from a Southern pine chip  
15 furnish. This pulp was prepared by a routine conventional kraft pulping process  
16 using methodology common to the industry. A sulfite process was used on a  
17 Southern pine chip furnish to characterize the behavior of fibers pulped from the  
18 same species via different pulping processes. This pulp was prepared by an acid  
19 bisulfite process (sulfite process) common to the industry. This pulp (Sample A-5i  
20 - Table I-5, Example 5) had a K Number of 34.

21

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1	EXAMPLE 5, TABLE I-5			
2	SAMPLE DESIGNATION	<u>A-5i</u>	<u>B-5i</u>	<u>C-5i</u>
3	SAMPLE DESCRIPTION			
4	Processing	Unbleached Pulp Before Cold Alkali Extraction	Unbleached Pulp After Cold Alkali Extraction	Unbleached Pulp After Cold Alkali Extraction
5				
6	Wood Species	Southern pine blend—————>		
7				
8	Pulping Process	Kraft—————>		
9	K Number, mL	30	—	—
10				
11	COLD ALKALI EXTRACTION	Not Used	Used	Used
12	Alkali Used		NaOH	NaOH
13	Solution Strength, %		15	18
14	Temperature, °C		30	30
15	Time, H:M		0:15	0:15
16	Consistency, %		3.0	3.0
17				
18	PULP ANALYTICAL PROPERTIES			
19	ISO Brightness, %	—	—	—
20				
21	MULTIPLE INSULT ABSORPTION TEST			
22	Absorption times, seconds			
23	1st insult	9.5	5.6	5.3
24	2nd insult	36.3	20.2	18.7
25	3rd insult	56.9	32.4	26.7
26				
27				
28				
29				

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## EXAMPLE 5, TABLE II-5

1				
2				
3	SAMPLE DESIGNATION	<u>A-5ii</u>	<u>B-5ii</u>	<u>C-5ii</u>
4	SAMPLE DESCRIPTION			
5	Processing	Unbleached Pulp Before Cold Alkali Extraction	Pulp After Cold Alkali Extraction	Pulp After Cold Alkali Extraction
6				
7	Wood Species	Southern pine blend----->		
8				
9	Pulping Process	Sulfite----->		
10	K Number, mL	34	-	-
11				
12	COLD ALKALI EXTRACTION	Not Used	Used	Used
13	Alkali Used		NaOH	NaOH
14	Solution Strength, %		15	18
15	Temperature, °C		30	30
16	Time, H:M		0:15	0:15
17	Consistency, %		3.0	3.0
18				
19	MULTIPLE INSULT ABSORPTION TEST			
20	Absorption times, seconds			
21	1st insult	13.2	6.9	5.1
22	2nd insult	36.3	24.5	18.6
23	3rd insult	45.1	32.3	23.2
24				

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1		<b>EXAMPLE 5, TABLE III-5</b>		
2	<b>SAMPLE DESIGNATION</b>	<u>A-5iii</u>	<u>B-5iii</u>	<u>C-5iii</u>
3	<b>SAMPLE DESCRIPTION</b>			
4	Processing	Bleached Pulp Before Cold Alkali Extraction	Bleached Pulp After Cold Alkali Extraction	Bleached Pulp After Cold Alkali Extraction
5				
6	Wood Species	Southern pine-blend—————>		
7				
8	Pulping Process	Kraft—————>		
9				
10	<b>COLD ALKALI EXTRACTION</b>	Not Used	Used	Used
11	Alkali Used		NaOH	NaOH
12	Solution Strength, %		15	18
13	Temperature, °C		30	30
14	Time, H:M		0:15	0:15
15	Consistency, %		3.0	3.0
16				
17	<b>PULP ANALYTICAL PROPERTIES</b>			
18	ISO Brightness, %	92	—	—
19				
20	<b>MULTIPLE INSULT ABSORPTION TEST</b>			
21	Absorption times, seconds			
22	1st insult	8.9	5.5	6.4
23	2nd insult	41.6	18.8	23.1
24	3rd insult	69.1	33.4	37.3
25				

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1                   Despite the different pulping processes, the Southern pine chip  
2 furnish yielded fibers having excellent absorption results after cold alkali extraction  
3 (Tables I-5 and II-5 or Example 5, the alkali used being sodium hydroxide). Each  
4 of the unbleached pulps was treated with a cold caustic solution of 15% and 18%  
5 NaOH (weight%). The cold caustic extraction was carried out as follows. Pulps of  
6 3% consistency [O.D. pulp weight/total weight (caustic solution + O.D. pulp) X  
7 100] were treated at about 30°C for about 15 minutes by stirring the suspension.  
8 For each different caustic solution treated sample, the absorbency was determined  
9 and compared for the respective Kraft and sulfite pulp. It is noted that extraction  
10 with 18% NaOH gave the best test results for each of the pulp stocks.

11                   For comparison, the unbleached kraft Southern pine pulp was  
12 bleached to an ISO brightness of 92% prior to applying the cold alkali extraction  
13 process (Sample A-5iii, Table III-5 Example 5). This pulp was the same pulp used  
14 as the starting material for bleaching in Example 5 (Sample A-5i, Table I-5).  
15 However, prior to cold caustic extraction it was bleached with the chemicals of  
16 chlorine, chlorine dioxide, hydrogen peroxide, oxygen and sodium hydroxide. The  
17 results obtained are shown in Table III-5 when following the same cold caustic  
18 extraction procedure as outlined above.

19                   From the comparison of the data in Tables I-5 and II-5 of this  
20 example, it is evident that for the unbleached pulp an increase in concentration of  
21 the cold caustic solution to 18% NaOH improved the absorption properties; for  
22 bleached pulp (Sample A-5iii, Table III-5) the higher concentration (*i.e.*, 18%  
23 NaOH) reduced the absorbency properties compared to extraction with 15%  
24 NaOH. However, note that the absorbency properties of the bleached pulp cold  
25 caustic extracted with 15% NaOH were distinctly better than those of the bleached  
26 pulp processed without any cold alkali extraction.

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- 1                    Example 6.    Prehydrolyzed Kraft Bleached Southern Pine Pulp
- 2                    In a manner similar to that used for Example 5, the absorption
- 3 properties were determined for a bleached, prehydrolyzed kraft pulp from Southern
- 4 pine wood. This pulp was prepared by a routine prehydrolyzed kraft pulping
- 5 process using methodology common to the industry and was bleached using the
- 6 chemicals of chlorine, chlorine dioxide, sodium hydroxide, and sodium
- 7 hypochlorite to an ISO Brightness of 86%. The K Number of the unbleached pulp
- 8 was about 18 mL.

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1	EXAMPLE 6, TABLE I-6			
2	SAMPLE DESIGNATION	<u>A-6i</u>	<u>B-6i</u>	<u>C-6i</u>
3	SAMPLE DESCRIPTION			
4	Processing	Bleached Pulp Before Cold Alkali Extraction	Bleached Pulp After Cold Alkali Extraction	Bleached Pulp After Cold Alkali Extraction
5				
6	Wood Species	Southern pine—————>		
7				
8	Pulping Process	Steam prehydrolyzed kraft—————>		
9				
10	COLD ALKALI EXTRACTION	Not Used	Used	Used
11	Alkali Used		NaOH	NaOH
12	Solution Strength, %		15	18
13	Temperature, °C		30	30
14	Time, H:M		0:15	0:15
15	Consistency, %		3.0	3.0
16				
17	PULP ANALYTICAL PROPERTIES			
18	ISO Brightness, %	86	—	—
19				
20	MULTIPLE INSULT ABSORPTION TEST			
21	Absorption times, seconds			
22	1st insult	6.7	4.9	6.2
23	2nd insult	46.6	19.6	25.5
24	3rd insult	77.2	32.3	41.9
25				

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1                   The absorption results after cold alkali extracting this pulp with 15%  
2   and 18% NaOH are shown in Table I-6, and confirm the effect caused by CCE  
3   treatment on the absorbency of the pulp. Again, this pine pulp subjected to  
4   extraction after bleaching with 15% NaOH (Sample A-6j, Table I-6, Example 6)  
5   gave better results than that extracted with 18% NaOH.

6                   In comparison to the absorption property data given in Example 5,  
7   Tables I-5 to III-5, the absorption test results for the prehydrolyzed kraft Southern  
8   pine fiber show that these are within a good range of absorbency despite having  
9   been bleached to this relatively high brightness prior to cold alkali extraction.

10                  Example 7.   Kraft Southern Pine Pulp: Unbleached K Number  
11   Interaction

12                  The accompanying Table I-7, Example 7, illustrates a comparative series of  
13   kraft Southern pine pulps where unbleached pulp K Number was varied (relative  
14   severity of pulping - low K Numbers indicate a more drastic pulping schedule with  
15   less lignin remaining in the pulp after pulping). In order to establish the desired  
16   pulp property regime and pulping procedures, the bleaching of all three  
17   unbleached stocks was carried out to equivalent brightness (92% ISO). The  
18   bleaching chemicals of chlorine, chlorine dioxide, hydrogen peroxide, oxygen and  
19   sodium hydroxide were used. It is noted that as unbleached K Number increased,  
20   resulting absorption properties after cold caustic extraction improved (lower  
21   absorption times are better).



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1                   Example 8.   Sitka Spruce Sulfite Pulp

2  
3                   In a manner similar to that used for the Southern pine chip furnish,  
4   spruce starting material was sulfite pulped. The sulfite process used was acid  
5   sulfite (also known as acid bisulfite) as in Example 5. The unbleached pulp was  
6   subjected to cold caustic extraction. Data for the cold caustic extracted pulps  
7   treated with 15% and 18% NaOH are shown in Table I-8, Example 8. It is noted  
8   that while not all starting pulps perform at the same level, nevertheless there was a  
9   significant improvement in absorbency after cold caustic solution treatment for  
10   each of the pulps obtained from this species. Both spruce pulp Samples B-8i and  
11   C-8i performed similarly (no significant difference between 15% NaOH and 18%  
12   NaOH extractions).

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EXAMPLE 8, TABLE I-8

1				
2				
3	SAMPLE DESIGNATION	<u>A-8i</u>	<u>B-8i</u>	<u>C-8i</u>
4	SAMPLE DESCRIPTION			
5	Processing	Unbleached Pulp Before Cold Alkali Extraction	Unbleached Pulp After Cold Alkali Extraction	Unbleached Pulp After Cold Alkali Extraction
6				
7	Wood Species	Sitka Spruce .....		>
8				
9	Pulping Process	Sulfite .....		>
10	K Number, mL	31 .....		>
11				
12	COLD ALKALI EXTRACTION	Not Used	Used	Used
13	Alkali Used		NaOH	NaOH
14	Solution Strength, %		15	18
15	Temperature, °C		30	30
16	Time, H:M		0:15	0:15
17	Consistency, %		3.0	3.0
18				
19	PULP ANALYTICAL PROPERTIES			
20	ISO Brightness, %	--	--	--
21				
22	MULTIPLE INSULT ABSORPTION TEST			
23	Absorption times, seconds			
24	1st insult	70.0	12.2	10.0
25	2nd insult	43.7	22.3	24.1
26	3rd insult	64.8	33.6	33.2
27				

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1                   Example 9.   Western Hemlock Sulfite Pulp  
2  
3                   This example illustrates the absorption properties obtained for a pulp  
4   made from a western hemlock chip furnish by a sulfite pulping process (acid  
5   sulfite). Cold caustic extraction of the bleached hemlock pulp (ISO Brightness =  
6   88%) again illustrates the improvement that results on cold alkali treatment: greater  
7   speed of absorbency of fibers produced by cold caustic solution extraction. The  
8   data are given in Table I-9, Example 9.

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1

## EXAMPLE 9, TABLE I-9

2	SAMPLE DESIGNATION	<u>A-9i</u>	<u>B-9i</u>	<u>C-9i</u>
3	SAMPLE DESCRIPTION			
4	Processing	Bleached Pulp Before Cold Alkali Extraction	Bleached Pulp After Cold Alkali Extraction	Bleached Pulp After Cold Alkali Extraction
5				
6	Wood Species	Western Hemlock—————>		
7				
8				
9	Pulping Process	Sulfite—————>		
10				
11	COLD ALKALI EXTRACTION	Not Used	Used	Used
12	Alkali Used		NaOH	NaOH
13	Solution Strength, %		15	18
14	Temperature, °C		30	30
15	Time, H:M		0:15	0:15
16	Consistency, %		3.0	3.0
17				
18	PULP ANALYTICAL PROPERTIES			
19	ISO Brightness, %	88	--	--
20				
21	MULTIPLE INSULT ABSORPTION TEST			
22	Absorption times, seconds			
23	1st insult	12.3	7.7	9.4
24	2nd insult	47.2	31.9	39.9
25	3rd insult	75.1	44.7	62.0
26				

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Example 10. BCTMP (bleached chemi-thermal mechanical pulp) and Cold Alkali Extraction

In this example, BCTMP (a bleached chemi-thermal mechanical pulp) commercially available from Tembec Co. was also extracted with 15% and 18% NaOH. The absorption test data (Table I-10, Example 10) show substantial improvement at the higher cold caustic solution strength. The K Number of this BCTMP pulp was 36 mL. Even higher caustic solution strengths (e.g., about 20%) may prove to be beneficial to absorbent property performance for fibers produced from this type of furnish via this pulping process. The wood furnish is a North American, eastern Canadian softwood. Description of chemi-thermal mechanical pulping and bleaching processes can be found in texts on pulping and bleaching.

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## EXAMPLE 10, TABLE I-10

1				
2				
3	SAMPLE DESIGNATION	<u>A-10i</u>	<u>B-10i</u>	<u>C-10i</u>
4	SAMPLE DESCRIPTION			
5	Processing	Bleached Pulp Before Cold Alkali Extraction	Unbleached Pulp After Cold Alkali Extraction	Unbleached Pulp After Cold Alkali Extraction
6				
7	Wood Species	Northern Softwood		>
8				
9	Pulping Process	Bleached Chemi-thermal mechanical		>
10	K Number, mL	36		>
11				
12	COLD ALKALI EXTRACTION	Not Used	Used	Used
13	Alkali Used		NaOH	NaOH
14	Solution Strength, %		15	18
15	Temperature, °C		30	30
16	Time, H:M		0:15	0:15
17	Consistency, %		3.0	3.0
18				
19	PULP ANALYTICAL PROPERTIES			
20	ISO Brightness, %	-	-	-
21				
22	MULTIPLE INSULT ABSORPTION TEST			
23	Absorption times, seconds			
24	1st insult	9.7	8.8	4.0
25	2nd insult	39.8	37.8	25.1
26	3rd insult	61.2	59.8	35.4
27				

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Example 11. Use of Cold Alkali Extraction on Semi-bleached, Lower Brightness Pulp

The strength of caustic solution required to achieve optimum absorption properties is related to the type of raw material and to pulping and bleaching steps used. In general, however, the less bleached (lower brightness) the pulp, the higher the concentration of NaOH solution required to achieve optimum properties. Also, the absorption properties attained are better when the pulp is less bleached prior to application of a cold caustic treatment. This is illustrated below in Table I-11, Example 11, where two Southern pine kraft pulps bleached to different brightness levels (ISO Brightness of 51 and 88, respectively) underwent cold alkali extraction with 15 and 18% NaOH solutions. The results for both of these pulps show that 15% NaOH gave the best overall absorption time results (third insult time is the most significant one) with the pulp of lower brightness (semi-bleached pulp) yielding superior properties.

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EXAMPLE 11, TABLE I-11

2	SAMPLE DESIGNATION	A-11i	B-11i	C-11i	D-11i
3	SAMPLE DESCRIPTION				
4	Processing	Semi-Bleached Pulp (ISO Brightness=51%) <sup>a</sup> After Cold Alkali Extraction—>		Bleached Pulp (ISO Brightness=88%) <sup>b</sup> After Cold Alkali Extraction—>	
5					
6	Wood Species	Southern pine blend—————>			
7					
8	Pulping Process	Kraft—————>			
9					
10	COLD ALKALI EXTRACTION	Used	Used	Used	Used
11	Alkali Used	NaOH—————>			
12	Solution Strength, %	15	18	15	18
13	Temperature, °C	30	30	30	30
14	Time, H:M	0:15	0:15	0:15	0:15
15	Consistency, %	3.0	3.0	3.0	3.0
16					
17	MULTIPLE INSULT ABSORPTION TEST				
18	Absorption times, seconds				
19	1st insult	5.0	3.8	6.2	4.9
20	2nd insult	22.1	21.2	22.2	24.8
21	3rd insult	25.1	36.7	33.9	38.6
22					
23					
24	(a)	Prepared by bleaching Sample A-11i of Example 11 (Table 11) with chlorine, chlorine dioxide,			
25		hydrogen peroxide, oxygen and sodium hydroxide to an ISO Brightness of 51.			
26	(b)	Prepared by bleaching Sample A-11i of Example 11 (Table 11) with chlorine, chlorine dioxide,			
27		hydrogen peroxide, oxygen and sodium hydroxide to an ISO Brightness of 88.			

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## EXAMPLE 11, TABLE II-11

1					
2					
3	SAMPLE DESIGNATION	A-11ii	B-11ii	C-11ii	D-11ii
4	SAMPLE DESCRIPTION				
5	Processing	Semi-Bleached Pulp (ISO Brightness=38%) <sup>a</sup> After Cold Alkali Extraction—————>		Semi-Bleached Pulp (ISO Brightness=44%) <sup>a</sup> After Cold Alkali Extraction—————>	
6					
7	Wood Species	Southern pine blend—————>			
8					
9	Pulping Process	Sulfite—————>			
10					
11	COLD ALKALI EXTRACTION	Used	Used	Used	Used
12	Alkali Used	NaOH—————>			
13	Solution Strength, %	15	18	15	18
14	Temperature, °C	30—————>			
15	Time, H:M	0:15—————>			
16	Consistency, %	3.0—————>			
17					
18	MULTIPLE INSULT ABSORPTION TEST				
19	Absorption times, seconds				
20	1st insult	4.5	4.5	5.8	4.7
21	2nd insult	24.5	18.8	24.1	20.2
22	3rd insult	36.4	30.2	41.1	31.2
23	(a)	Prepared by bleaching Sample A-5ii of Example 5 (Table II-5) with chlorine dioxide,			
24		hydrogen peroxide and sodium hydroxide.			

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1 All of the Samples A-11i through D-11i (Table I-11, Example 11)  
2 were prepared by bleaching the 30 K Number Southern pine kraft pulp discussed  
3 in Example 5 (Sample A-5i, Table I-5) to the brightness level indicated, and then  
4 cold caustic extracted under the conditions indicated. Note that in the unbleached  
5 state, 18% NaOH resulted in better absorption properties and that these absorption  
6 times were better than those associated with semi-bleached pulps described in  
7 Table I-11, Example 11.

8 When the 34 K Number unbleached Southern pine sulfite pulp of  
9 Example 5, Table II-5 (Sample A-5ii) was semi-bleached to ISO Brightness levels of  
10 38 and 44, respectively, prior to cold caustic extraction with 15 and 18% NaOH,  
11 18% NaOH was still required to give optimum absorption properties. The results of  
12 this work are seen in Table II-11, Example 11. Note that less bleached pulp (ISO  
13 Brightness = 38%) still gives the best results when extracted with 18% NaOH  
14 (Sample B-11ii versus Sample D-11ii); the results, however, are not as good as  
15 those observed by 18% NaOH extraction of the unbleached pulp itself with 18%  
16 NaOH (see results for Sample C-5ii, Table II-5, Example 5).

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1                    Example 12. Position of Cold Alkali Extraction  
2    in a Multistage Bleach Sequence  
3  
4                    The benefits of cold caustic extraction in improving absorbency  
5 occurs regardless of where it is applied in the bleaching sequence (e.g., at the  
6 beginning, the middle, or at the very end). In the same multi-stage bleaching  
7 sequence to prepare high brightness pulps there is even some indication that  
8 when the same quantity of chemicals are used, there may be improved absorption  
9 properties by applying the stage in the middle of the sequence. Such an example  
10 is now presented in which the only bleaching variable, in a 5-stage sequence to  
11 prepare a fully bleached pulp from the same unbleached stock, was the position of  
12 the CCE stage in the sequence; 15% NaOH solution strength was used.

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EXAMPLE 12, TABLE I-12

SAMPLE DESIGNATION	A-12i	B-12i	C-12i
SAMPLE DESCRIPTION			
Processing	Fully Bleached Pulp (5 Bleach Stages) with Cold Alkali Extraction in Stage 1	Fully Bleached Pulp (5 Bleach Stages) with Cold Alkali Extraction in Stage 3	Fully Bleached Pulp (5 Bleach Stages) with Cold Alkali Extraction in Stage 5
Wood Species	Southern pine		
Pulping Process	Prehydrolyzed kraft		
COLD ALKALI EXTRACTION	Used		
Alkali Used	NaOH		
Solution Strength, %	15		
Temperature, °C	30		
Time, H:M	0:15		
Consistency, %	3.0		
PULP ANALYTICAL PROPERTIES			
ISO Brightness, %	84	87	84
MULTIPLE INSULT ABSORPTION TEST			
Absorption times, seconds			
1st insult	17.7	14.4	19.2
2nd insult	44.5	31.2	43.4
3rd insult	67.7	57.2	70.4

The results shown above (Table II-12) indicate that when CCE was used in the middle of the sequence (stage 3), the absorption time results were clearly better (Sample B-12i) than when CCE was used in the 1st or 5th stages. It was interesting to note that ISO Brightness of the fully bleached pulp (Sample B-12i) was also improved relative to the other two (i.e., 87 versus 84%).

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1                    Example 13. Kraft Pulping Reject Material

2                    In an effort to look at pulp that is even "rawer" or less pulped than  
3                    what normally occurs in conventional full chemical pulping processes, some  
4                    "knots" resulting from a conventional kraft cook of Southern pine chip furnish  
5                    were cold caustic extracted with 18% NaOH. "Knots" essentially represent  
6                    pulping reject materials that are poorly cooked (relatively large in size unlike shives  
7                    and separable from the resulting pulped fibers via equipment loosely termed  
8                    "knotters"). It was necessary to first defiber these knots in a Waring blender and  
9                    to flat screen this defibered material to remove reject material still remaining non-  
10                    defibered prior to cold alkali extraction with 18% NaOH. The fibers had a very high  
11                    K Number (>50).

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## EXAMPLE 13, TABLE I-13

1		
2		
3	SAMPLE DESIGNATION	<u>A-13i</u>
4	SAMPLE DESCRIPTION	
5	Processing	Defiberized Knots (Rejects) After Cold Alkali Extraction
6		
7	Wood Species	Southern pine blend
8		
9	Pulping Process	Kraft
10	K Number, mL (80 mL test)	>50
11		
12	COLD ALKALI EXTRACTION	Used
13	Alkali Used	NaOH
14	Solution Strength, %	18
15	Temperature, °C	30
16	Time, H:M	0:15
17	Consistency, %	3.0
18		
19	PULP ANALYTICAL PROPERTIES	
20	ISO Brightness, %	-
21		
22	MULTIPLE INSULT ABSORPTION TEST	
23	Absorption times, seconds	
24	1st insult	5.4
25	2nd insult	13.9
26	3rd insult	25.9
27		

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1                   The multiple insult absorption test results shown in Table I-13,  
2   Example 13 indicate that these type of fibers after cold alkali extraction exhibit  
3   good absorption times. The absorption times are equivalent to those of the cold  
4   alkali extraction 30 K Number unbleached kraft Southern pine pulp (Sample C-5i,  
5   Table I-5, Example 5) and to those of the cold alkali extracted 34 K Number  
6   unbleached Southern pine sulfite pulp (Sample C-5i, Table II-5, Example 5),  
7   despite the raw material being essentially a waste material. A cold caustic  
8   extraction would add significant value in turning this waste fiber into a viable  
9   absorbent product.

10                It is believed that similar results would be obtained by stopping the  
11   initial kraft pulping process at a point(s) corresponding to less delignification  
12   overall and combining mechanical defiberization and screening steps prior to cold  
13   alkali extraction and/or any bleaching desired to increase brightness (*i.e.*, semi-  
14   chemical rather than full chemical pulping).

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1                    Example 14. Alkali Source Other Than Sodium Hydroxide:  
2                    Kraft White Liquor

3  
4                    A potential source of sodium hydroxide within a kraft pulp mill is  
5 white liquor used in the kraft pulping process. White liquor is a mixture of sodium  
6 hydroxide and sodium sulfide. A suggestion is to carry out cold alkali extraction  
7 using the alkali present in white liquor (WL) as the source of NaOH; it is also  
8 possible that the sodium sulfide present in the white liquor will have some positive  
9 benefits. In Table I-14, Example 14, are presented results of extraction of an  
10 unbleached Southern pine kraft pulp with 9-18% NaOH in which some or almost all  
11 of the NaOH requirements in the cold caustic extraction came from the white liquor  
12 itself (the contribution of sodium sulfide to alkalinity was ignored). For comparative  
13 purposes, cold caustic extractions with 9, 15 and 18% NaOH solutions were also  
14 carried out as controls (Samples F-14i, G-14i, and H-14i, Table I-14).

EXAMPLE 14, TABLE I-14

SAMPLE DESIGNATION SAMPLE DESCRIPTION Processing	A-14i	B-14i	C-14i	D-14i	E-14i	F-14i	G-14i	H-14i
	Unbleached Pulp Before Cold Alkali Extraction	Unbleached Pulp After Cold Alkali Extraction with White Liquor (WL) <sup>a</sup>	Unbleached Pulp After Cold Alkali Extraction	Unbleached Pulp After Cold Alkali Extraction	Unbleached Pulp After Cold Alkali Extraction (Control)	Unbleached Pulp After Cold Alkali Extraction (Control)	Unbleached Pulp After Cold Alkali Extraction (Control)	Unbleached Pulp After Cold Alkali Extraction (Control)
Wood Species	Southern pine							
Pulping Process	Kraft							
K Number, mL	18.7							
COLD ALKALI EXTRACTION Alkali Used	Not Used (Control)	Used NaOH (96% from WL)	NaOH (67% from WL)	NaOH (48% from WL)	NaOH (37% from WL)	NaOH	NaOH	NaOH
Solution Strength, %		9	12	15	18	9	15	18
Temperature, °C		30						
Time, H:M		0:15						
Consistency, %		3.0						
MULTIPLE INSULT ABSORPTION TEST								
Absorption times, seconds								
1st insult	7.0	6.7	6.5	6.5	6.7	8.0	7.0	5.9
2nd insult	37.2	25.3	23.7	25.0	24.3	30.4	26.1	26.8
3rd insult	60.7	42.2	37.3	38.9	37.6	50.5	35.9	37.0
(a) Effective alkali (NaOH wt. %) = 9.29.								

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1           The use of white liquor to supply alkali was equivalent to the use of  
2   sodium hydroxide at 15 and 18% NaOH solution strength (compare Samples C-14i  
3   and G-14i, D-14i and H-14i, Table I-14, Example 14) in achieving improved  
4   absorption properties relative to those of the non-cold alkali extracted unbleached  
5   Southern pine kraft pulp. At 9% solution strength, the use of white liquor appeared  
6   to result in some improvement over the use of NaOH alone (compare Sample B-  
7   14i to F-14i, Table I-14i, Example 14). It is believed that at even lower alkali  
8   solution strengths white liquor may also result in advantages over the use of  
9   sodium hydroxide alone.

10           Example 15. Use of Hemi Caustic for Cold Alkali Extraction

11           In using sodium hydroxide for cold alkali extraction, a caustic solution is  
12   obtained which contains some organic material removed or "extracted" from the  
13   pulp. This type of caustic solution is termed "hemi caustic". The organic materials  
14   solubilized during the cold alkali reaction with the pulp are considered to be  
15   predominantly hemicellulosic materials (hemicelluloses are non-cellulosic  
16   carbohydrate materials composed of xylose, mannose, or mixtures of these  
17   monomers with glucose, etc. rather than the 100% glucose monomer of cellulose).  
18   For a fibrous end-use pulp, cold alkali extraction may also remove some of these  
19   hemicelluloses or other organics. However, the desired end-result is not  
20   chemically purer pulp fibers. Purity is required in dissolving pulps because these  
21   pulps must function as chemical feedstocks in chemical end-use processes (esters  
22   such as acetates, butyrates and nitrates, ethers, regenerated cellulose, etc.). For a  
23   fibrous end-use application, the end result desired from the use of cold alkali  
24   extraction is that the fibers produced exhibit improved performance as fibers -- as  
25   fluffed fibers in absorbent products, etc.; such appreciation of the desired end  
26   result heretofore has not been recognized or known.

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## EXAMPLE 15, TABLE I-15

1				
2				
3	SAMPLE DESIGNATION	<u>A-15i</u>	<u>B-15i</u>	<u>C-15i</u>
4	SAMPLE DESCRIPTION			
5				
6	Processing	Standard Process (Bleached Pulp prior to Cold Alkali Extraction)	Cold Alkali Extraction (Bleached Pulp subsequent to Cold Alkali Extraction—>	
7				
8	Wood Species	Southern pine blend—————>		
9				
10	Pulping Process	Kraft—————>		
11	K Number, mL	17.4—————>		
12				
13	COLD ALKALI EXTRACTION	Not Used	Used—————>	
14	Alkali Used	(Control	100% NaOH	100% hemi caustic
15	Solution Strength, %		15—————>	
16	Temperature, °C		30—————>	
17	Time, H:M		0:15—————>	
18	Consistency, %		3—————>	
19				
20	PULP ANALYTICAL PROPERTIES			
21	ISO Brightness, %	84.4	85.5	84.4
22				
23	MULTIPLE INSULT ABSORPTION TEST			
24	Absorption times, seconds			
25	1st insult	4.7	4.9	4.9
26	2nd insult	25.6	18.8	20.4
27	3rd insult	44.8	29.9	36.6
28				
29				

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1                   The data presented in Example 15, Table I-15 show that hemi  
2   caustic (*i.e.*, caustic separated from the pulp after reaction under an initial pure  
3   sodium hydroxide cold caustic extraction) can be reused to supply the alkali  
4   source for subsequent cold caustic extractions for pulps with improvements in  
5   absorbency properties. Absorption times are improved relative to the non-cold  
6   caustic extracted bleached Southern pine kraft pulp when the alkali source used  
7   was either sodium hydroxide or hemi caustic. The hemi caustic solution used was  
8   at 24.5% sodium hydroxide by weight and contained 2.9% "hemicellulose"  
9   material. The improvements when the hemi caustic was used were not as great as  
10   those when pure sodium hydroxide was used. However, it is expected that some  
11   modification of the cold caustic extraction conditions (for example, increasing the  
12   solution strength when hemi caustic is used) would make the effects of both types  
13   of caustic equivalent.

14                It would also follow that other alkali sources when used in initially  
15   contacting the pulp in a cold alkali extraction could be reused in subsequent  
16   extraction for these types of fibrous end-use non-dissolving pulps.

17                In the above Examples the cold caustic solution treatment or cold  
18   caustic extraction of the pulp was typically at the indicated solution strength, at the  
19   indicated consistency, for the indicated time at the indicated temperature, followed  
20   by a fresh water rinse, an acid wash (typically a sulfuric acid solution at pH of  
21   about 3) and a final fresh water rinse.

22                Other test data that were obtained also indicate that for the entire  
23   range of concentration of the cold caustic solution, the concentration may range  
24   between about 5% to 25% and higher but 13% to 18% gave the best results for the  
25   various pulp starting materials utilized for acquisition layers, *i.e.* intensive, fast  
26   absorption uses. A suitable concentration is dependent on the relative severity of

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1 bleaching with the more severely bleached pulps requiring a milder treatment. For  
2 absorbency improvements in general and also for improving yields and other fluff  
3 pulp properties lower concentrations of caustic may be used i.e. to about 5%. The  
4 versatility of the process has also been demonstrated for a variety of pulp source  
5 materials.

6           As illustrated in the examples above the cold caustic solution has  
7 been a sodium hydroxide solution, but other alkali materials may be used. Other  
8 alkali materials such as potassium hydroxide etc. may be used but at a severe  
9 economic penalty such that their use is prohibitive.

10           Likewise, a combination of sodium hydroxide solution and a water  
11 soluble, non-toxic glycol, (e.g., propylene glycol solution) might also be used, but  
12 the added cost is less justified for this large volume bulk product.

13           In describing the regime for the acceptable starting pulps and  
14 process conditions for CCE treatment, this regime may be characterized as  
15 follows: for fast absorbency improvements such as measured by the insult tests,  
16 especially the third insult; the K Number related to absorbency and severity of  
17 pulping; the severity of pulping which may be avoided when practicing the present  
18 utilization of various pulps in bleached and unbleached conditions; enhanced  
19 yields of accept fibers upon fluffing; advantageous use of reject materials; use of  
20 mill by-products; swing capability to insert in the bleaching treatment steps the  
21 CCE step in any bleaching sequence; fluff pulp properties not requiring debonders,  
22 i.e. without additives, etc. etc. Such improvements especially in combination with  
23 each other have heretofore not been recognized, known or practiced for fluff pulps  
24 and thus have not shown the way to the unique combination(s) of properties  
25 described above.

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1                   The basis weight of acquisition layers in current products ranges  
2   from 75 to 200 g/m<sup>2</sup>. As an example, the acquisition layer 12 shown in Figure 2 is  
3   an air laid fluff web of 200 g/m<sup>2</sup>. This web is separated from the absorbent core  
4   13 by a layer of conventionally wet-laid tissue paper 13. The core may be  
5   wrapped in such tissue paper. The absorbent core is a mixture of cellulose fiber,  
6   fluffed and airlaid with super absorbent polymer (SAP) available from commercial  
7   sources. The basis weight is about 500-700 g/m<sup>2</sup>. There is a moisture proof  
8   polymer backsheet 16 below the core of 0.5 mil. Wet laid sheets may also be  
9   used.

10                  The above described examples, embodiments, and comparisons are  
11   intended to illustrate the various aspects of the invention without limitation of same  
12   but the appended claims and elements thereof including reasonable equivalents  
13   for these are to define the metes and bounds of the invention.

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1 WHAT IS CLAIMED:

2 Claim 1. In a process for improving the characteristics of a  
3 pulp useful for making a fluff pulp or a pulp for absorbency intensive applications  
4 the improvement comprising:

5 treating a pulp at a temperature of up to about 60°C, in a  
6 suspension, with an alkali solution of a concentration from about 2% to about 25%  
7 by weight, for a treatment time sufficient to obtain a pulp of improved absorbency  
8 characteristics, and

9 recovering said thus treated pulp from said suspension suitable for  
10 intensive absorbency and fluff pulp use applications.

11 Claim 2. In a method for improving absorbency of pulps and  
12 increasing yields of accept fibers upon fluffing thereof the improvement  
13 comprising:

14 subjecting a pulp fiber suspension at a temperature of less than  
15 about 45°C, in a fiber suspension from about 2% up to about 25% consistency, to  
16 a caustic solution of a concentration of about 5% to 25% by weight for a time  
17 sufficient to improve the absorbency characteristics of a pulp material resulting  
18 from such treatment.

19 Claim 3. In a process for improving pulp properties of pulps  
20 useful as fluff pulps the improvement comprising:

21 subjecting a pulp fiber suspension at a temperature of less than  
22 about 45°C, in a fiber suspension from about 2% up to about 25% consistency, to  
23 a caustic solution of a concentration of about 5% to 25% by weight for a time  
24 sufficient to improve the absorbency characteristics of a pulp material resulting  
25 from such treatment.

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1                   Claim 4.       The process as defined in Claim 3, and wherein the  
2   temperature of said pulp fiber suspension is less than about 40°C, the fiber  
3   suspension is from about 2% to 10% consistency, and the caustic solution is at a  
4   concentration from about 5% to about 18% by weight.

5                   Claim 5.       The process as defined in Claim 3, wherein the  
6   concentration of said caustic solution is between 13% and 15% by weight.

7                   Claim 6.       The process as defined in Claim 3 wherein the  
8   concentration of said caustic solution is between 5% and 10% by weight.

9                   Claim 7.       The process as defined in Claim 3, wherein said pulp  
10   is from a pulp source starting material of Southern pine, White pine, Western  
11   hemlock, Sitka spruce, Caribbean pine, Douglas fir or other softwoods or mixtures  
12   of same.

13                  Claim 8.       The process as defined in Claim 3, wherein said pulp  
14   is from a pulp source starting material of gums, oaks, eucalyptus, poplar, beech,  
15   aspen or bagasse.

16                  Claim 9.       The process as defined in Claim 3, wherein the  
17   temperature of said caustic treatment is about 30°C and a time of treatment is from  
18   about 5 minutes to about one hour.

19                  Claim 10.     In a process for improving the fast absorbency  
20   characteristics of a pulp useful in absorbency intensive applications the  
21   improvement comprising:

22                         treating pulp at a temperature of less than about 40°C in a  
23   suspension with a caustic solution of a concentration from about 13% to about  
24   18% by weight, said concentration being dependent on the amount of lignin  
25   remaining in the pulp, as measured by the K number and a severity of pulping of

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1 said pulp, for a treatment time sufficient to obtain a pulp of improved absorbency,  
2 and

3 recovering the thus treated pulp from said suspension suitable for  
4 intensive absorbency and fluff pulp use applications.

5 Claim 11. The process as defined in Claim 10, wherein said pulp  
6 is an unbleached pulp with a K Number of 8 or above before the same is treated  
7 with caustic solution.

8 Claim 12. The process as defined in Claim 10, wherein said pulp  
9 is partially bleached before treatment of same with said caustic solution.

10 Claim 13. The process defined in Claim 10, wherein the pulp is  
11 a chemical-mechanical pulp or organic solvent obtained pulp.

12 Claim 14. The process as defined in Claim 12, wherein a  
13 bleached pulp is a kraft process pulp, before said pulp is treated with said cold  
14 caustic solution.

15 Claim 15. The process as defined in Claim 12, wherein said  
16 bleached pulp is treated with a caustic solution of a concentration inversely  
17 proportional to a severity of bleaching to which said pulp had been subjected and  
18 wherein said pulp maintains its improved absorbency characteristics upon  
19 rewetting.

20 Claim 16. The process as defined in Claim 10, wherein said  
21 absorbency intensive application is for an acquisition layer for a baby diaper.

22 Claim 17. The process as defined in Claim 10, wherein a pulp  
23 source starting material is a pulp derived from a softwood.

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1                   Claim 18.     The process as defined in Claim 10, wherein said pulp  
2 is bleached prior to its treatment with a caustic solution to an ISO brightness  
3 percentage of about 25 and higher.

4                   Claim 19.     The process as defined in Claim 10, wherein said pulp  
5 is treated with a caustic solution in a suspension of about 3% by weight (O.D.)  
6 pulp at a temperature from about 25°C to about 40°C for a period of time sufficient  
7 to improve said absorbency for said pulp.

8                   Claim 20.     The process as defined in Claim 10, wherein pulps of  
9 low K Number of at least 10 are treated with a caustic solution of a concentration  
10 of up to about 15%.

11                  Claim 21.     The process as defined in Claim 20, wherein the  
12 concentration of said caustic solution is between 13% and 15% by weight.

13                  Claim 22.     The process as defined in Claim 10 wherein said pulp  
14 is from a pulp source starting material of Southern pine, White pine, Western  
15 hemlock, a Sitka spruce, Caribbean pine, a Douglas fir or mixtures of same.

16                  Claim 23.     The process as defined in Claim 10, wherein said pulp  
17 is from a pulp source starting material of gums, oaks, eucalyptus, poplar, beech,  
18 aspen, bagasse or mixtures of same.

19                  Claim 24.     The process as defined in Claim 10, wherein the  
20 temperature of said caustic treatment is about 30°C and a time of treatment is from  
21 about 5 minutes to about one hour.

22                  Claim 25.     In a process of constructing an absorbent device  
23 having an outer acquisition layer and an inner absorbent core element, the  
24 improved process comprising:

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1                   pulping a pulp source starting material to a preselected K Number of  
2   about 8 and above to obtain a pulp with substantially said K Number and wherein  
3   said pulp is optionally bleached;

4                   treating said pulp at a temperature of less than about 45°C in a  
5   suspension with a caustic solution of a concentration from about 5% to about 25%  
6   by weight, with a treatment time sufficient to obtain a pulp of improved absorbency  
7   values, and recovering thus treated pulp from said suspension suitable for  
8   absorbency applications in said device;

9                   sheeting and drying said pulp into a sheet of a basis weight from  
10   200 to 800 grams per meter squared; and

11                  converting said sheet to an outer layer for said diaper on at least  
12   one surface of a core element of said device or a core element for said device.

13                  Claim 26.     The process as defined in Claim 25, wherein said  
14   core element is composed at least in part of improved absorbency pulp obtained  
15   as defined in Claim 25 derived from Southern pine pulp.

16                  Claim 27.     The process as defined in Claim 25, wherein the  
17   device is a baby diaper.

18                  Claim 28.     The process as defined in Claim 25, wherein the  
19   device is a catamenial device.

20                  Claim 29.     The process as defined in Claim 25, wherein the  
21   device is an incontinence device.

22                  Claim 30.     The process as defined in Claim 25, wherein an  
23   absorbent pulp component is of a pulp obtained from hardwood pulp.

24                  Claim 31.     The process as defined in Claim 25, wherein the  
25   absorbent pulp is of a pulp from Western hemlock.

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1                   Claim 32.     The process as defined in Claim 25, wherein sheeting  
2 and drying said pulp is after flash drying and collecting of said pulp.

3                   Claim 33.     An improved pulp for an absorbent device comprised  
4 of at least an acquisition layer element wherein said layer is of a pulp as defined in  
5 Claim 10.

6                   Claim 34.     An improved absorbency material comprised of a  
7 cellulosic fibrous material wherein said cellulosic fibrous material has been  
8 obtained by pulping a cellulosic source material which has an unbleached pulp K  
9 Number of at least 10 and wherein said cellulosic fibrous material is a cold caustic  
10 solution treated material at a treatment temperature of less than about 40°C, in a  
11 suspension of 2% to 15%, with said cold caustic solution being at a concentration  
12 of from about 5% to 25% by weight.

13                  Claim 35.     The improved absorbency material as defined in  
14 Claim 34 wherein the cellulosic fibrous material subsequent to cold caustic  
15 treatment has been mechanically treated.

16                  Claim 36.     The improved absorbency material as defined in  
17 Claim 34 wherein the cellulosic fibrous material subsequent to cold caustic  
18 treatment has been beaten.

19                  Claim 37.     The improved absorbency material as defined in  
20 Claim 34 wherein the unbleached pulp K Number for same is at least about 20 and  
21 above.

22                  Claim 38.     The improved absorbency material as defined in  
23 Claim 34 above wherein the same is incorporated into a baby diaper, a catamenial  
24 device, an incontinence device, a towel or a tissue in sheet form.

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1                   Claim 39.     In a process for improving the absorbency of a  
2   cellulosic material in a fibrous form of said cellulosic material wherein said material  
3   is useful in absorbency applications, the improvement comprising:  
4                   treating said cellulosic material at a temperature of less than about  
5   45°C, in suspension, with a caustic solution of a concentration from about 5% to  
6   about 10% by weight, said concentration being dependent on the process  
7   employed, wood species used and/or on the amount of lignin remaining in said  
8   cellulosic material as measured by a K Number measurement, wherein said caustic  
9   solution is in contact with said cellulosic material for a treatment time sufficient to  
10   obtain a cellulosic material of improved absorbency values, and  
11                  recovering thus treated cellulosic material from said suspension  
12   suitable for absorbency applications.

13                Claim 40.     The process as defined in Claim 39, wherein said  
14   cellulosic material is an unbleached pulp with a K Number of at least about 8 or  
15   above before treatment of same with said caustic solution.

16                Claim 41.     The process as defined in Claim 39, wherein said  
17   cellulosic material is a partially bleached pulp before treatment of same with said  
18   caustic solution.

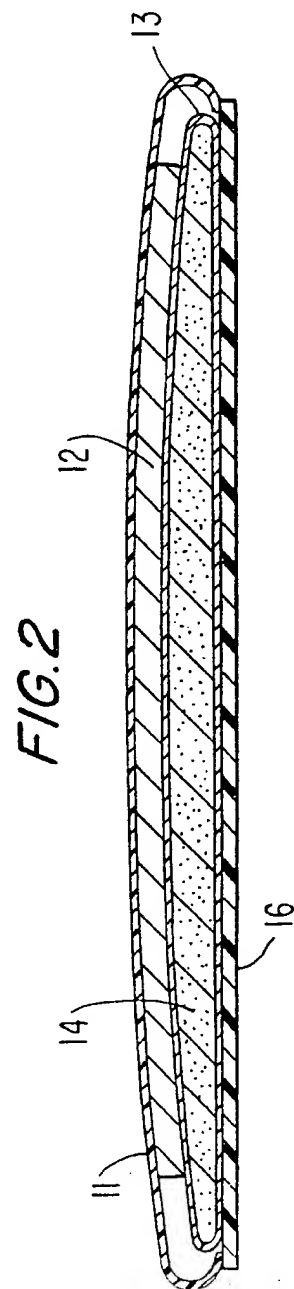
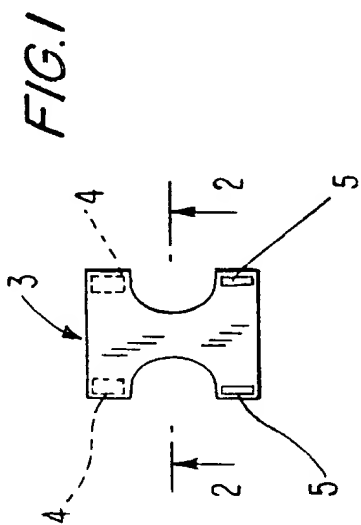
19                Claim 42.     The process as defined in Claim 39, wherein the  
20   cellulosic material is a bleached pulp, before treatment of said pulp with said cold  
21   caustic solution.

22                Claim 43.     The process as defined in Claim 41, wherein said  
23   bleached pulp is treated with a caustic solution of a concentration inversely  
24   proportional to a severity of bleaching to which said pulp had been subjected and

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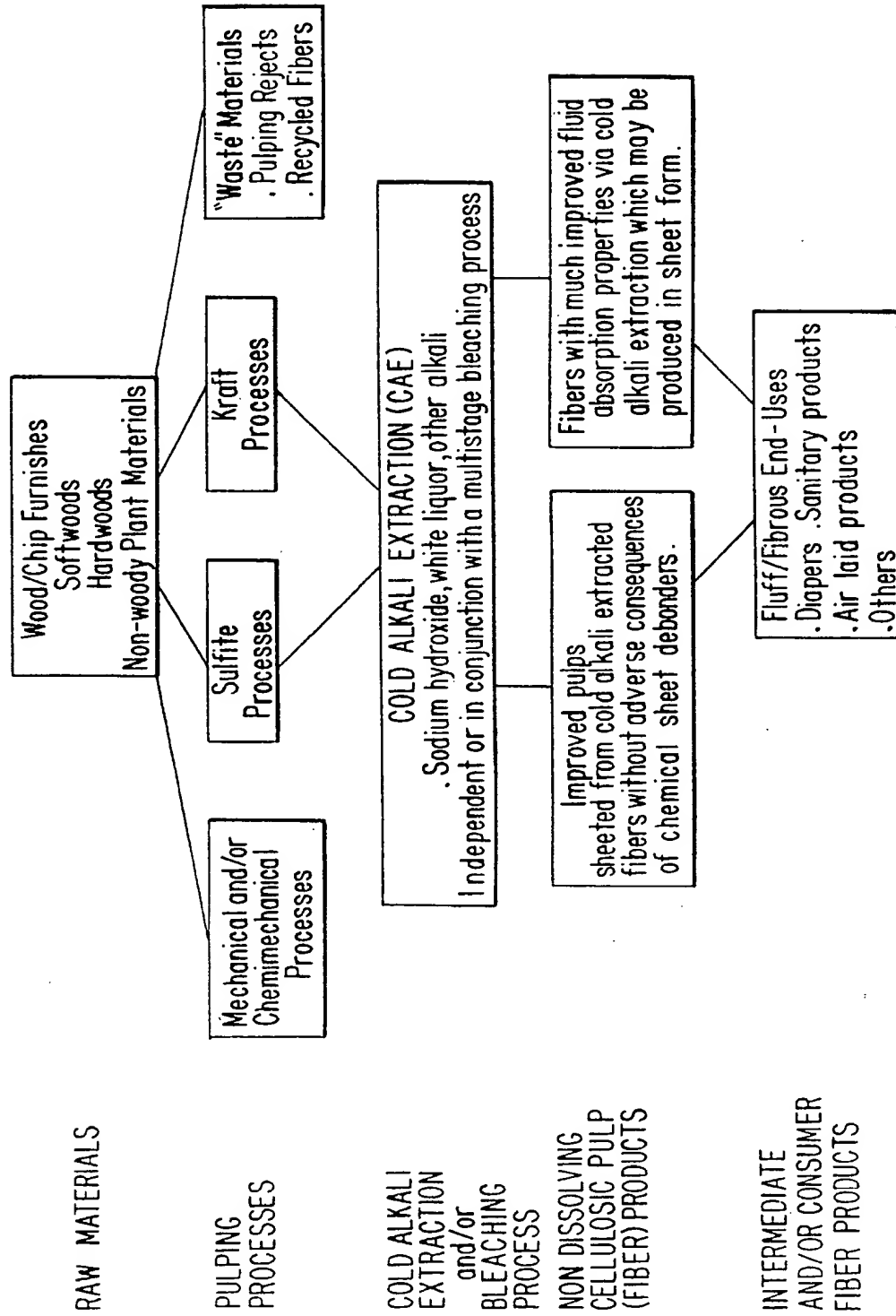
- 1 . wherein said pulp maintains its improved absorbency characteristics upon
- 2 rewetting.

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FIG. 3



## INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US95/00862

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :D21C 3/02, 3/26

US CL. :162/90, 111

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 162/8, 24. 82, 83, 90, 111

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched  
NONE

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)  
NONE

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US, A, 1,913,283 (McCORMICK ET AL) 6 June 1933, page 2, lines 22-23, page 2, lines 68-74, and page 5, lines 3-10.	1-43
Y	US, A, 4,689,118 (MAKOU ET AL) 25 August 1987, see col.1, lines 7-12.	16, 25-32 and 38

☐ Further documents are listed in the continuation of Box C.

☐ See patent family annex.

* Special categories of cited documents:	*T	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
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Date of the actual completion of the international search

14 APRIL 1995

Date of mailing of the international search report

03 MAY 1995

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